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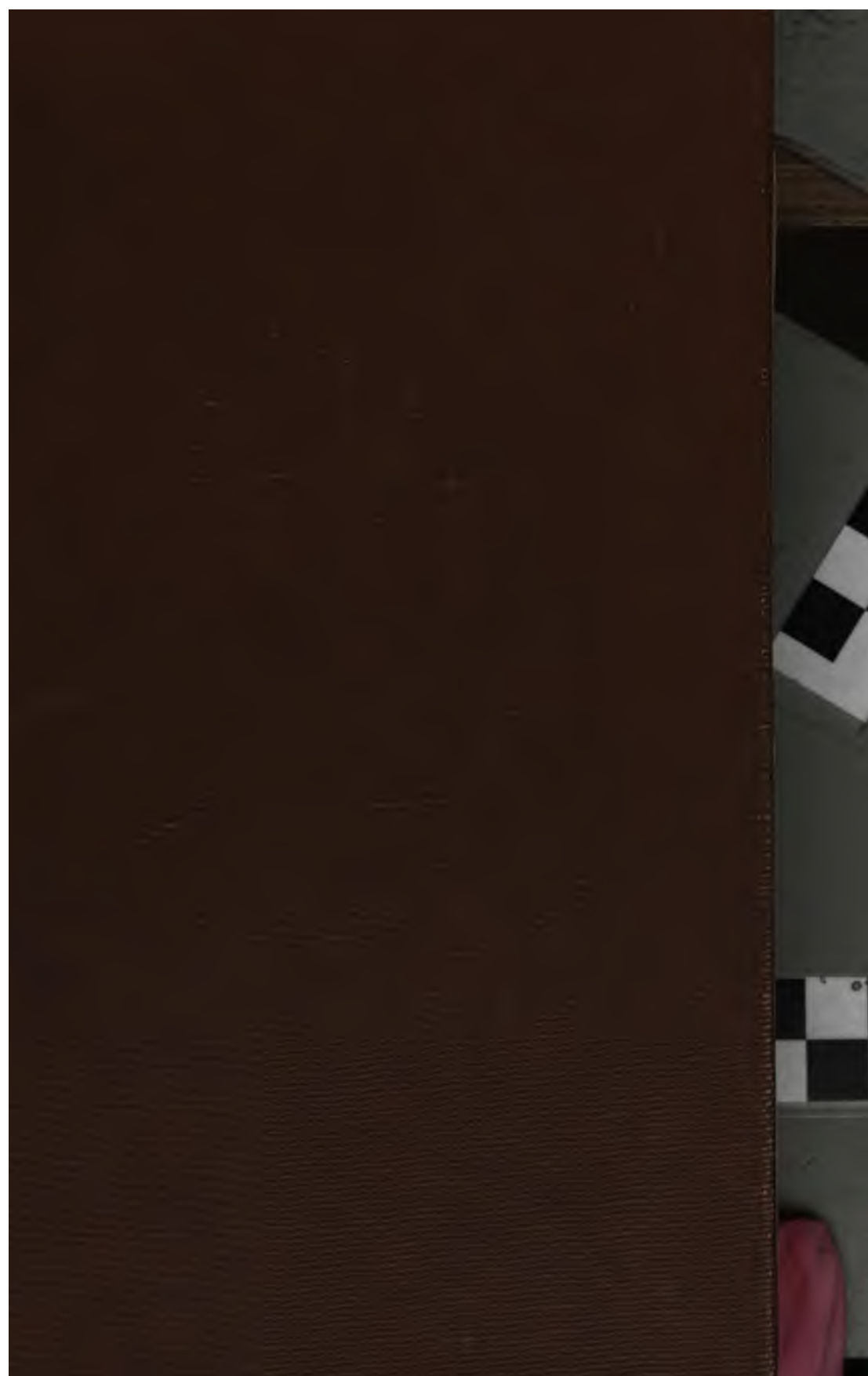
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DEPARTMENT OF THE INTERIOR  
UNITED STATES GEOLOGICAL SURVEY

CHARLES D. WALCOTT, DIRECTOR

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THE  
AREAS OF THE UNITED STATES, THE  
STATES, AND THE TERRITORIES

BY

HENRY GANNETT



WASHINGTON  
GOVERNMENT PRINTING OFFICE

1906

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## ILLUSTRATION.

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<b>PLATE I. Map of the United States, defining the adopted limits of the gross area .....</b>	<b>5</b>
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# THE AREAS OF THE UNITED STATES, THE STATES, AND THE TERRITORIES.

By HENRY GANNETT.

In 1881 the Census Office published in an extra bulletin tables showing the areas of the United States and of the several States and Territories. The measurements and computations upon which these tables were based were made with care, according to methods described in the bulletin, and the results probably represented the areas as closely as they could be determined from the maps and charts of that time. In 1899 the General Land Office printed similar tables, which were also prepared with great care and thoroughness.

The tables presented in these two publications differ from each other. Most of the differences are trifling, amounting to only a few square miles or a small fraction of 1 per cent, being well within the limits of error of the planimeter and of the maps used. Some of them, however, are considerable, and a few are explained by the fact that more recent maps, which changed the position of boundaries between States, had been used by the Land Office, and its measurement was, therefore, more nearly correct. Other discrepancies arose from differences in determining the coast lines; as an example of this, the Land Office measurement of the State of Washington included half of the Strait of Juan de Fuca, while that of the Census Office did not, involving a difference in area of 1,500 square miles.

Realizing the desirability of the Government issuing but one statement of areas of the States and Territories, the offices concerned, through their representatives, Mr. Frank Bond, chief draftsman of the General Land Office, Mr. C. S. Sloane, geographer of the Census Office, and myself, representing the Geological Survey, have been at work for several months in the endeavor to come to an agreement on these figures, and in the course of our discussion many new measurements have been made from the most recent and best maps. An agreement has been reached, the results of which are set forth in the accompanying table.



By this adjustment the area of the United States proper is increased over the Census Office figures by 1,188 square miles, or about three one-hundredths of 1 per cent.

The question, "What constitutes the area of the United States?" is by no means a simple one. Jurisdiction extends to a line 3 nautical miles from the shore, but this strip of sea can not properly be regarded as a part of the country. Supposing our country to be restricted to the sea and lake coast, there remains a question regarding the bays and estuaries. To what extent should the coast line be followed strictly, and where should we begin to jump across the indentations made by the sea? In this matter one can only follow his own judgment, making in each case as natural a decision as possible, as no definite criterion can be established. This applies also to those States bordering the sea and the Great Lakes.

The method of measurement may be characterized in a few words. The areas of all square degrees included entirely within a State or the United States are taken from tables of such areas. Where a square degree is crossed by a boundary line, so that only part of it is included, both the part included and that excluded are measured from the best maps by planimeter, and the correctness of the measurement is tested by comparing their sum with the tabular area of the square degree.

The entire boundary of the United States has now been mapped or charted with accuracy, so that, aside from uncertainties due to the interpretation of the coast line, there can be little or no question as to the correctness of the measurement of the country as a whole. The situation as regards the individual States is not so satisfactory, however, although the boundaries of most of them are now well mapped. Certain States are bounded by parallels and meridians, and the ascertainment of their areas is merely a matter of reference to tables of areas of square degrees, assuming that the boundary lines were accurately run and marked in accordance with the statute. The boundaries of a few of the States, however, have not been accurately mapped, and it is probable that changes will be made in the computed areas of these States.

The area of Alaska, measured in this office from the large Coast Survey chart, is subject to considerable modification in the future as the position of the coast line becomes better known.

The area given for the Philippine Islands is that determined by the Coast Survey of that archipelago, prepared at the instance of the Philippine Census and published in that report. It also is subject to modification as accurate charts of the archipelago are made.

The areas of Hawaii and Porto Rico are probably subject to only slight change, as the charts from which they were measured are quite accurate.

The areas given for the other small possessions of the United States—Guam, Samoa, and the Panama Canal strip—will probably be changed in the future as their limits become more correctly defined.

*Areas, in square miles, of the States and Territories.*

State or Territory.	Land surface.	Water surface.	Total area.
Alabama .....	51, 279	719	51, 998
Arizona .....	113, 840	116	113, 956
Arkansas .....	52, 525	810	53, 335
California .....	156, 092	2, 205	158, 297
Colorado .....	103, 658	290	103, 948
Connecticut .....	4, 820	145	4, 965
Delaware .....	1, 965	405	2, 370
District of Columbia .....	60	10	70
Florida .....	54, 861	3, 805	58, 666
Georgia .....	58, 725	540	59, 265
Idaho .....	83, 779	534	84, 313
Illinois .....	56, 002	663	56, 665
Indiana .....	35, 885	469	36, 354
Indian Territory .....	30, 790	419	31, 209
Iowa .....	55, 586	561	56, 147
Kansas .....	81, 774	384	82, 158
Kentucky .....	40, 181	417	40, 598
Louisiana .....	45, 409	3, 097	48, 506
Maine .....	29, 895	3, 145	33, 040
Maryland .....	9, 941	2, 386	12, 327
Massachusetts .....	8, 039	227	8, 266
Michigan .....	57, 480	500	57, 980
Minnesota .....	80, 858	3, 824	84, 682
Mississippi .....	46, 362	503	46, 865
Missouri .....	68, 727	693	69, 420
Montana .....	145, 776	796	146, 572
Nebraska .....	76, 808	712	77, 520
Nevada .....	109, 821	869	110, 690
New Hampshire .....	9, 031	310	9, 341
New Jersey .....	7, 514	710	8, 224
New Mexico .....	122, 503	131	122, 634
New York .....	47, 654	1, 550	49, 204
North Carolina .....	48, 740	3, 686	52, 426
North Dakota .....	70, 183	654	70, 837
Ohio .....	40, 740	300	41, 040
Oklahoma Territory .....	38, 624	224	38, 848

## AREAS OF STATES AND TERRITORIES.

*Areas, in square miles, of the States and Territories—Continued.*

State or Territory.	Land surface.	Water surface.	Total area.
Oregon.....	95,607	1,092	96,699
Pennsylvania.....	44,832	294	45,126
Rhode Island.....	1,067	181	1,248
South Carolina.....	30,495	494	30,989
South Dakota.....	76,868	747	77,615
Tennessee.....	41,687	335	42,022
Texas.....	262,398	3,498	265,896
Utah.....	82,184	2,806	84,990
Vermont.....	9,124	440	9,564
Virginia.....	40,262	2,365	42,627
Washington.....	66,836	2,291	69,127
West Virginia.....	24,022	148	24,170
Wisconsin.....	55,256	810	56,066
Wyoming.....	97,594	320	97,914
	2,974,159	52,630	3,026,789
Alaska.....			590,884
Guam.....			210
Hawaii.....			6,449
Panama Canal Zone.....			474
Philippine Islands.....			115,026
Porto Rico.....			3,435
Tutuila Group, Samoa.....			77
Total.....			3,743,344

Owing to their location adjoining the Great Lakes, the States enumerated below contain approximately an additional number of square miles as follows:

Illinois.....	1,674 square miles of Lake Michigan.
Indiana.....	230 square miles of Lake Michigan.
Michigan.....	16,653 square miles of Lake Superior.
	12,922 square miles of Lake Michigan.
	9,925 square miles of Lake Huron.
	460 square miles of Lakes St. Clair and Erie.
Minnesota.....	2,514 square miles of Lake Superior.
New York.....	3,140 square miles of Lakes Ontario and Erie.
Ohio.....	3,443 square miles of Lake Erie.
Pennsylvania.....	891 square miles of Lake Erie.
Wisconsin.....	2,378 square miles of Lake Superior.
	7,500 square miles of Lake Michigan.

In addition to the water areas noted above, California claims jurisdiction over all Pacific waters lying within 3 English miles of her coast; Oregon claims jurisdiction over a similar strip of the Pacific Ocean 1 marine league in width between latitude 42° N. and the mouth of the Columbia River; and Texas claims jurisdiction over a strip of Gulf water 3 leagues in width, adjacent to her coast between the Rio Grande and the Sabine River.



## CLASSIFICATION OF THE PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY.

[Bulletin No. 302.]

The publications of the United States Geological Survey consist of (1) Annual Reports; (2) Monographs; (3) Professional Papers; (4) Bulletins; (5) Mineral Resources; (6) Water-Supply and Irrigation Papers; (7) Topographic Atlas of United States—folios and separate sheets thereof; (8) Geologic Atlas of United States—folios thereof. The classes numbered 2, 7, and 8 are sold at cost of publication; the others are distributed free. A circular giving complete lists may be had on application.

Most of the above publications may be obtained or consulted in the following ways:

1. A limited number are delivered to the Director of the Survey, from whom they may be obtained, free of charge (except classes 2, 7, and 8), on application.

2. A certain number are delivered to Senators and Representatives in Congress, for distribution.

3. Other copies are deposited with the Superintendent of Documents, Washington, D. C., from whom they may be had at prices slightly above cost.

4. Copies of all Government publications are furnished to the principal public libraries in the large cities throughout the United States, where they may be consulted by those interested.

The Professional Papers, Bulletins, and Water-Supply Papers treat of a variety of subjects, and the total number issued is large. They have therefore been classified into the following series: A, Economic geology; B, Descriptive geology; C, Systematic geology and paleontology; D, Petrography and mineralogy; E, Chemistry and physics; F, Geography; G, Miscellaneous; H, Forestry; I, Irrigation; J, Water storage; K, Pumping water; L, Quality of water; M, General hydrographic investigations; N, Waterpower; O, Underground waters; P, Hydrographic progress reports. This paper is the fifty-third in Series F, the complete list of which follows (all are bulletins thus far except Professional Paper 45):

### SERIES F. GEOGRAPHY.

5. Dictionary of altitudes in United States, by Henry Gannett. 1884. 325 pp. (Out of stock; see Bulletin 274.)
6. Elevations in Dominion of Canada, by J. W. Spencer. 1884. 43 pp. (Out of stock.)
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76. Dictionary of altitudes in United States (second edition), by Henry Gannett. 1891. 393 pp. (Out of stock; see Bulletin 274.)
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117. Geographic dictionary of Connecticut, by Henry Gannett. 1894. 67 pp.
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## II

## SERIES LIST.

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226. Boundaries of the United States and of the several States and Territories, with an outline of the history of all important changes of territory (third edition), by Henry Gannett. 1904. 145 pp., 54 pls.
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233. Gazetteer of West Virginia, by Henry Gannett. 1904. 164 pp.
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248. Gazetteer of Indian Territory, by Henry Gannett. 1904. 70 pp.
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- Professional Paper 45. The geography and geology of Alaska, a summary of existing knowledge, by A. H. Brooks, with a section on climate, by Cleveland Abbe, jr., and a topographic map and description thereof, by R. U. Goode. 1906. 327 pp., 34 pls.
271. A dictionary of altitudes in the United States (fourth edition), compiled by Henry Gannett. 1906. 1072 pp.
276. Results of primary triangulation and primary traverse, fiscal year 1904-5, by S. S. Gannett. 1905. 263 pp., 1 pl.
281. Results of spirit leveling in the State of New York for the years 1896 to 1905, inclusive, by S. S. Gannett and D. H. Baldwin. 1906. 112 pp.
288. Results of spirit leveling in Pennsylvania for the years 1899 to 1905, inclusive, by S. S. Gannett and D. H. Baldwin. 1906. 62 pp.
291. Gazetteer of Colorado, by Henry Gannett. 1906. 185 pp.
299. Geographic dictionary of Alaska, by Marcus Baker; second edition, prepared by James McCormick. 1906. — pp.
302. Areas of the United States, the States, and the Territories, by Henry Gannett. 1906. 9 pp., 1 pl.

Correspondence should be addressed to

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# PRELIMINARY ACCOUNT OF GOLDFIELD, BULLFROG, AND OTHER MINING DISTRICTS IN SOUTHERN NEVADA.

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By **FREDERICK LESLIE RANSOME.**

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## INTRODUCTION.

The following report aims to give in brief form such advance results as are available from a detailed investigation of the geology of the Goldfield and Bullfrog districts carried out during the autumn and winter of 1905-6. Complete reports on these districts are now in preparation and will be published as soon as possible. In the present bulletin are included also such reconnaissance notes as were made on outlying districts visited in connection with the principal work of the field season. Messrs. W. H. Emmons and G. H. Garrey, who had efficiently assisted in the work throughout the season, were instructed at its close to visit the Manhattan district, mainly for the purpose of ascertaining the area that should be included in future mapping and study of this new gold field. Their notes on the geology of the district are published as a separate section in this report.

During the summer and autumn of 1905 a topographic map, covering 8,500 square miles of southern Nevada and including the Goldfield and Bullfrog districts, was made by Messrs. R. H. Chapman and B. D. Stewart, topographers. A copy of this map accompanies this report (Pl. I). Mr. S. H. Ball was attached to Mr. Chapman's party as geologist, and the results of his reconnaissance of the whole region, made under the writer's general supervision, will be published as a separate bulletin (No. 308).

## GOLDFIELD DISTRICT.

### INTRODUCTION.

In June, 1903, Mr. J. E. Spurr paid a hasty visit to Goldfield, then known as the Grandpa district. Some indications of ore were known at this time, but no important ore bodies had been found. Mr. Spurr



published a brief note<sup>a</sup> on the geology of the district, which was supplemented by a fuller statement,<sup>b</sup> made after a second visit in November, 1904.

During the summer of 1905 a topographic map on a scale of 2,000 feet to the inch and covering approximately 31½ square miles of the district was made by Mr. William Stranahan, topographer, and, with this map as a base, detailed geological work was begun in September of the same year by the writer, assisted by Messrs. W. H. Emmons and G. H. Garrey. Field work was completed in December.

The following account has been prepared to meet the demand for prompt information regarding a new and important district. It is not intended as a complete or final report on the geology and ores of Goldfield, but merely as a provisional advance statement, which will be amplified and modified when all the material gathered during the field work shall have been fully studied. Experience has shown that preliminary accounts of new mining districts are usually of value, but the benefit to the mining community may be gained at some cost to an author's professional reputation unless the object and limitations of such a preliminary outline are clearly understood by the reader. Therefore in this case it should be understood that the right to revise statements and conclusions is distinctly reserved, and when the final report appears it should entirely supersede the earlier publication.

#### HISTORY.

The discovery of Tonopah in 1900 marked, for Nevada, the beginning of a new era in its mining industry, which in the years following the decline of the Comstock had sunk to a very low ebb. As the size and richness of the Tonopah bonanzas became known new stimulus was given to prospecting and the desert ranges of southwestern Nevada were more thoroughly searched than ever before. Among the dozens of new camps that have come into existence within the last few years as a result of this activity, Goldfield, situated in Esmeralda County, 24 miles nearly due south of Tonopah (see fig. 1 and Pl. I, in pocket), has attained chief importance.

"Float" gold was found late in 1902 by Harry Stimler and William Marsh on Columbia Mountain and, after seeking in vain for the source of this gold, these early prospectors worked northward and on December 2 located the Sandstorm lode. A brief period of excitement followed the discovery of gold, but, as no important ore bodies were immediately uncovered, interest flagged and only a few determined men remained in the district. Among these were A. D. Myers and R. C. Hart, who, on May 24, 1903, located the Combination lode

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<sup>a</sup> Bull. U. S. Geol. Survey No. 225, 1904, pp. 118-119.

<sup>b</sup> Bull. U. S. Geol. Survey No. 260, 1905, pp. 132-139.

and began at once to develop their property. In October of the same year, after the existence of ore had been proved by a short tunnel, the ground was bonded to the Combination Mines Company and in the following December this company began the shipment of ore.

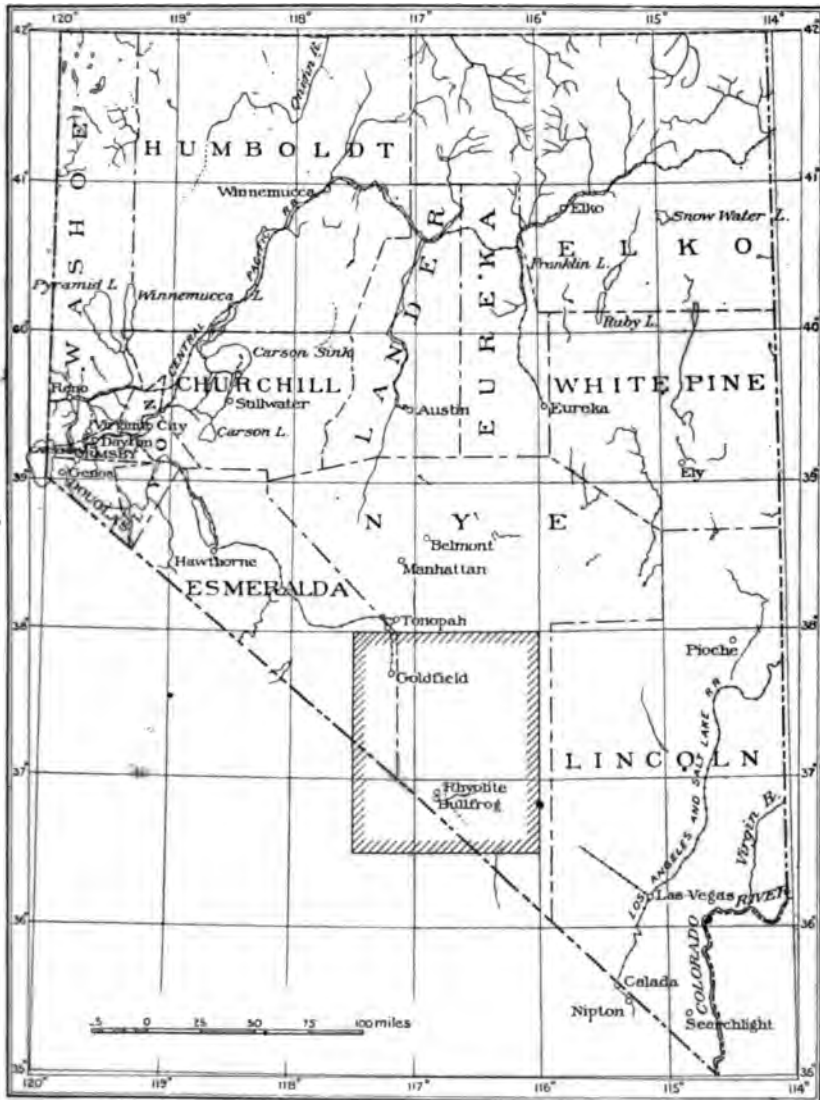


FIG. 1.—Index map of Nevada.

Prospecting was actively resumed over the district in January and February of 1904, and before midsummer rich ore was being shipped from the Jumbo, Florence, and January mines.

With the exception of the Combination, all the mines which have attained any importance were first opened and developed by the leasing system, and so near the surface and so rich were many of the ore bodies that numbers of the successful lessees made ample fortunes in a few months or even in a few weeks. The royalties usually exacted ranged from 20 to 30 per cent of the net value of the ore extracted. At one time there were six sets of lessees working through as many shafts on the Jumbo lode. The blocks were from 100 to 300 feet in length, the most productive and best known being that generally referred to as the Bowes-Kernick lease.

The daily output from all of these workings in August, 1904, was estimated at \$10,000, and the total to the expiration of the leases, early in 1905, amounted to considerably more than \$1,000,000. Another famous lease was the Sweeny lease, on the Florence mine, which expired on January 15, 1905, after a production amounting to \$650,000. The Patrick lease on the January mine, probably the first important lease granted in the camp, produced about \$500,000. Later, and in some respects more sensational than the leases just mentioned, was that granted to B. J. Reilly and associates on No. 1 block of the Florence ground. This lease, after having been successively abandoned by two earlier holders, was taken up by Reilly and his partners in April, 1905. Ore was found in the following June, but it was not until August that the true richness of the body became apparent. In the short time then available before the expiration of the term, on October 19, 1905, nearly all of the ore which the lease produced was mined and hoisted to the surface. The royalties paid to the Florence-Goldfield Mining Company represent a total output of \$475,000. Most accounts, however, give the production as \$750,000. Some of this ore was of extraordinary richness, a lot of about 4½ tons, taken out from the bottom of the workings during the last days of the lease, averaging, according to Mr. Frank Oliver, superintendent of the Florence mine, between \$6,000 and \$7,000 per ton.

Early in 1905 the district probably contained fully 5,000 people, over two-thirds of whom were in the town of Goldfield. Late in the year, owing to the attractions of Manhattan and other recently opened camps and to a decreasing activity in prospecting, in leasing, and in the various more or less speculative transactions so characteristic of a new mining district, the population had somewhat declined.

The completion of a railroad from Tonopah to Goldfield in September, 1904, gave the district a much-needed improvement in transportation facilities, although freight charges are still maintained at unduly high figures. Another important event of the year was the discovery of a large body of rich ore in the Reddy mine, which had not previously been a productive property.

## PRODUCTION.

In 1904 the Goldfield district produced over \$2,300,000. Statistics for the year 1905 are not yet available, but it is doubtful whether the output for the second year of activity will exceed \$2,500,000.

## TOPOGRAPHY.

The hills in the vicinity of Goldfield form an irregular group (see Pl. I), having no definite trend and belonging to no recognized mountain range. On the north, east, and south these hills slope down to broad undrained sterile basins, each with its characteristic central playa—a gleaming level plain incrustated with salts, except when an occasional shower has spread a film of water over its surface. The average altitude of the playa floors above sea level in this region is about 5,000 feet. Such a basin, approximately 10 miles in width, separates the Goldfield Hills from Stonewall Mountain, a prominent landmark southeast of the district. A low, broad pass between this basin and one to the north of it connects the Goldfield Hills with the northwest end of the Cactus Range, Cactus Peak being 20 miles east-northeast from Goldfield. A similar inconspicuous divide, about 12 miles north-northeast of Goldfield, forms a slight connection between the Goldfield Hills and the hills of the Southern Klondike and Tonopah districts.

West of town a basaltic mesa, at this latitude about 4 miles wide, but becoming much broader toward the south, stretches to the eastern base of Montezuma Peak, which is composed in part of rocks similar to those of the Goldfield district. The old mining settlement of Montezuma lies near the summit of the peak, which is 8,426 feet in height and 7 miles a little south of west from the town of Goldfield.

Goldfield is situated on the southern rim of one of the typical desert basins of the region, which merges through a low pass 22 miles north of town into the still larger basin west of Tonopah. This pass, through which the railroad runs from Tonopah to Goldfield, is between the Lone Mountain Range on the west and the Tonopah Hills on the east.

The portion of the Goldfield Hills which is particularly described in the present paper and which will for convenience be hereafter referred to as the Goldfield district is shown in the accompanying map (Pl. II), a much reduced and simplified copy of the geological map that will be published with the final report. The relief in this area ranges from 5,400 feet in its northwest corner to 6,635 feet on the summit of Preble Mountain, near the southeast corner. The culminating peak of the Goldfield Hills, however, is just east of the area represented in the map and attains an elevation of nearly 7,000 feet. A large part of the district may be described as a rolling plateau (see

fig. 2), ranging from 5,700 to 5,800 feet in general elevation and surmounted by many irregularly distributed peaks and knobs, which Preble, Blackcap, Vindicator, Banner, and Columbia mountains are conspicuous examples. None of these hills is a volcanic crater, as has been asserted in some descriptions of the district, nor are they due to local volcanic upheaval. They owe their height above the surrounding country merely to the fact that they are composed of material somewhat harder or more durable than the average rock of the region and have thus succumbed more slowly to the general erosion that has carved the existing relief from a gently domed volcanic series. The superior resistance of the hills is in some cases due to the original character of the rock of which they are composed; in other cases, to a capping of young and fresh lava, but by far the greater number of the smaller knobs that stud the district, of which Black Butte is an excellent example, are prominent because the rock has been hardened by local silicification in a manner that will be described in connection with the ore deposits.

The fact that much of the district is a rolling plain diversified by rocky knobs has an important bearing in a region where detritus tends to accumulate on all but the steepest slopes. In the Tonopah district, and especially in the Bullfrog district, there is generally a fairly definite line between steep, bare, rocky hills and the gentle alluvial slopes at their base. At Goldfield, however, the general gradients, as may be seen from fig. 2, are so slight that detritus accumulates over much of the surface and interferes seriously with attempts to determine the distribution and structure of the underlying rocks.

#### GENERAL GEOLOGY.

##### INTRODUCTION.

The geological structure of Goldfield compared with that of most districts in which deposits of the precious metals are found is simple. The district is essentially a low, domical uplift of Tertiary lavas and tuffs resting upon a foundation of ancient granitic and metamorphic rocks. The erosion of this flat dome has exposed

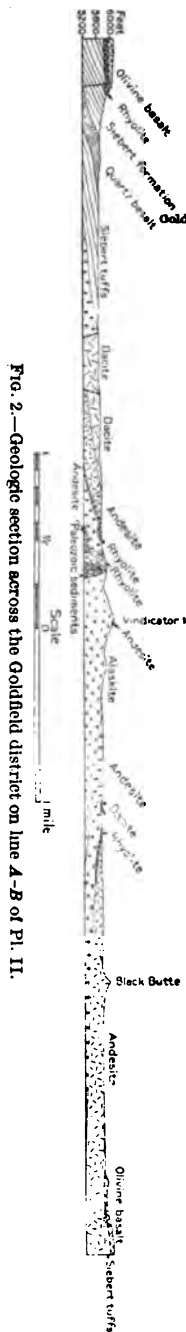


FIG. 2.—Geologic section across the Goldfield district on line A-B of Pl. II.

1

2



pre-Tertiary rocks at a number of places in the central part of the district, and these outcrops are surrounded by wide concentric zones of successively younger formations. But while it is not difficult to recognize the broad, bare outlines of the structure, this general idea requires modification in many particulars before it can be accepted as a satisfactory conception of the geology of the region, and it is in the study of these essential details rather than in complexity of structure that the investigation of the district offers real difficulties. One of these obstacles, the extensive cloaking of the surface by gravelly alluvium or "wash," has already been referred to. Owing to the prevalence of this material the contacts between the various underlying rocks are seldom well exposed and are usually completely covered. While it would be a simple matter to map this detritus wherever it is thick enough to conceal the rock beneath it, the map thus made would be less useful to the miner than one in which an attempt has been made to show the distribution of the possibly ore-bearing rocks, observed facts being supplemented wherever necessary by inferences based upon all available data. In accordance with this general principle the areas of alluvium have been entirely omitted from the preliminary map forming Pl. II and will be reduced to a minimum in the complete and final map. That some adjustment of geological boundaries will be demanded when mining operations determine the positions of contacts now concealed is of course to be expected; but it is deemed more important to present a helpful interpretation of hidden structure than to be content merely with cautious and unsuggestive adherence to visible facts.

Another obstacle to accurate and successful study of the region is the intensity, widespread distribution, and erratic character of the alteration that has affected rocks, some of which, even in a fresh state, could be distinguished from one another only by refined petrographic methods.

#### PRE-TERTIARY ROCKS.

The oldest rocks exposed in the district are found on Columbia and Vindicator mountains, and are particularly noticeable on the southern slope of the former peak as conspicuous dark patches visible from Columbia or Goldfield. Originally thin-bedded limestones or calcareous shales, with subordinate beds of sandstone, the formation is now much contorted and metamorphosed. The calcareous beds have been changed to compact aggregates of quartz (jasperoid) crowded with microscopic black particles, probably carbonaceous, and the sandstone to hard, vitreous quartzite.

The metamorphism is apparently due to the intrusion of these sediments by masses of a granitic rock composed essentially of quartz and orthoclase, a type for which Mr. Spurr has proposed the name *alaskite*. This rock is well exposed on Columbia Mountain and makes up a large



part of Vindicator Mountain. Smaller areas occur in the vicinity of Banner Mountain. Most of the alaskite contains a very little black mica and some plagioclase. In most exposures the mica is decomposed and scarcely noticeable, but northeast of the summit of Vindicator Mountain the alaskite mass grades into a biotite granite.

The age of the alaskite and intruded sediments, beyond the fact that both are pre-Tertiary, can not be determined from any evidence available in the Goldfield district. Mr. Spurr,<sup>a</sup> whose wide experience in southern Nevada renders his opinion of special weight, considers that the metamorphic sediments may well be Paleozoic and that the alaskite was intruded in post-Jurassic time. Mr. S. H. Ball,<sup>b</sup> who recently made a reconnaissance over a large area around the Goldfield district, found Cambrian fossils in beds which he is inclined to correlate with those exposed in Columbia and Vindicator mountains.

It is certain that the intrusion of the alaskite was followed by a long interval during which the region was eroded to the surface of moderate relief subsequently covered by the irruption of Tertiary lavas.

#### TERTIARY VOLCANIC ROCKS.

The sequence of Tertiary eruptions as shown by the rocks of the Goldfield district was as follows, the oldest rock being at the bottom of the column:

Olivine basalt; flow and small intrusive bodies.  
 Breccia and conglomerate.  
 Rhyolite; flow.  
 Lake beds.  
 Quartz-bearing basalt; flow intercalated in lake sediments.  
 Lake beds.  
 Latite and rhyolite; flows.  
 Dacite; sheetlike and irregular intrusions, possibly in part a flow.  
 Andesite; flows with some tuff and small intrusive bodies.  
 Rhyolite; intrusive masses.  
 Rhyolite; flows and tuffs.  
 Andesite; flow.

The older or first andesite, as may be seen from Pl. II, occupies an area a little over 2 miles in diameter in the center of the district, and a smaller area south of Columbia. Its characteristic features may be well seen in the vicinity of McLaughlan's well, on Banner Mountain, and on the southern edge of the town of Columbia. This rock has not been found in a fresh condition. Its color is usually dull purplish or greenish gray. A spotted or mottled appearance, due to flow-brecciation, is rather characteristic and is well shown on a small hill half a mile north of the summit of Banner Mountain. The freshest obtain-

<sup>a</sup> The ores of Goldfield, Nev.: Bull. U. S. Geol. Survey No. 260, 1905, p. 133.

<sup>b</sup> Oral communication.

able specimens of the first andesite show phenocrysts of feldspar, hornblende, and mica in a compact, streaky groundmass that was evidently once a glass with well-marked flowage lines. All the phenocrysts have the dullness that indicates more or less thorough alteration and the original glassy base is now devitrified. As a rule, the first andesite, when it has not been subjected to the intense local alteration connected with ore deposition, may be distinguished from the later andesitic rocks by the absence of fresh phenocrysts and the peculiar streakiness of its groundmass, which resembles the groundmass of a rhyolite rather than that of most andesites.

The rock is apparently a flow resting upon a clean and rather uneven surface of alaskite and Paleozoic sediments. Its thickness is not known, but is probably very variable, as on Columbia Mountain the succeeding rhyolite rests directly upon the alaskite. The maximum thickness is tentatively estimated as at least 400 feet, and it may be considerably more.

Overlying the first andesite, or resting on the pre-Tertiary rocks where the andesite is absent, is a series of thin glassy rhyolite flows which is best developed in the rounded white hills northwest of Columbia Mountain. This rhyolite is associated with pale-greenish tuffs of mingled andesitic and rhyolitic character and is cut by masses of intrusive rhyolite. All these rocks are shown together in Pl. II, although they will be distinguished on the final map. The effusive rhyolite is usually nearly white, shows conspicuous flow lamination, and weathers in thin, shelly flakes. This is the rock of the Sandstorm mine and of the hills lying west of the road from the mine to Columbia. It all shows some devitrification and silicification, particularly in the vicinity of the ore deposits. Certain varieties or portions of this rhyolite acquired by flowage a very thin lamination, causing the rock when somewhat weathered to closely resemble indurated clay shale. This appearance is very deceptive, as may be seen at the Tonopah Club or Conqueror mine. The town of Diamondfield is partly underlain by this shalelike rhyolite, and small residual patches of similar material are scattered over the surface of the first andesite. The rhyolite appears to have been thickest in the vicinity of Columbia Mountain and to thin out to the southeast. None of it has been found southeast of McLaughlan's well.

The intrusive rhyolite occurs on the summit of Columbia Mountain, where it has locally a sheetlike form and constitutes the ridge extending north of the mountain to the Adams mine. It is intrusive into the shaly rhyolite at the Conqueror mine, and another small mass cuts the first andesite, alaskite, and pre-Tertiary block jasperoid on the west slope of Vindicator Mountain. Thin sections indicate that some of this intrusive rhyolite occurs also on the east slope of Vindicator Mountain, but owing to the intensity of the alteration in this vicinity

it could not be separated in the field from the silicified first andesite. The tuffs associated with these early rhyolitic eruptions may be best examined along the southwest base of Columbia Mountain and directly west of the Kendall mine. Underground they are exposed in the west workings of the 100-foot level of this mine.

After the eruptions of the earlier rhyolites came the eruptions of the second andesitic period, which covered the region with successive flows of andesite, accompanied by subordinate layers of andesitic tuff. The products of these eruptions constitute the most extensive formation in the district. (See Pl. II.) The total thickness of this deposit, as of most of the other volcanic formations in this district, can not be determined, owing to the slight deformation of the rocks, their poor exposures, the comparatively slight topographic relief, and the absence of persistent datum planes, such as may usually be found in stratified rocks. It probably lies between 600 and 1,000 feet; but this is confessedly little more than a guess and is likely to be under rather than above the truth.

The petrographic study of the andesites has not been finished. They contain in general phenocrysts of labradorite feldspar, hornblende, augite, orthorhombic pyroxene, and occasionally biotite in groundmasses ranging from hyalopilitic to vitrophyric in texture. The hornblende exhibits various stages of magmatic resorption, the abundance of the augite being in rough inverse proportion to the diminution of the hornblende by this process. The second andesite undoubtedly includes more than one flow, but it is quite impracticable in this field to discriminate individual flows in mapping the whole area. Preliminary examination of over 200 specimens and thin sections of the andesites indicates that any division based on the predominance of certain ferromagnesian constituents—for example, augite andesite or hornblende andesite—would be artificial. In other words, the relative proportions of these minerals appear to vary in any one flow. Moreover, as the first andesite and pre-Tertiary rocks are cut by dikes and small intrusive bodies of the second andesite, the latter formation as a whole is probably a volcanic complex, in which are involved intrusive bodies, as well as flows and beds of tuff and breccia.

The dacite, whose eruption followed the second andesitic period, is in many respects the most interesting rock in the district and is certainly the most important from the economic standpoint. It occupies, as may be seen by reference to Pl. II, a considerable area east of Goldfield and Columbia and is the principal country rock of the Combination, January, Jumbo, Redtop, and Florence mines. The surface at the main Florence shaft is the second andesite, and it was at first supposed that the ore also occurred in this rock. Microscopic study shows, however, that most of the altered country rock of the Florence

below the zone of complete oxidation, although finer grained than the typical dacite, is really a variety of that rock, and consequently that the shaft penetrates the flat-lying andesite and enters the dacite. This mass of dacite appears to be an intrusive sheet dipping gently to the west and south and cut off to the west by faulting. Its structural relations will be more fully described in subsequent pages.

A larger body of dacite extends southward from Tognoni Springs and forms some of the high hills just east of the mapped portion of the district. This mass, as may be seen from Pl. II, makes up a large part of Preble Mountain. Smaller bodies of dacite occur southeast of Black Butte and in the central part of the district.

The dacite, of which typical and fairly fresh specimens can be collected on the little hill 500 feet northeast of the Transformer station, has the appearance of an andesite. It is generally more conspicuously porphyritic than the andesites of the district, but its essential distinction from them is the presence of phenocrysts of quartz. These are always rounded by magmatic corrosion and as a rule are neither very numerous nor very conspicuous. The rock consists of labradorite feldspar, hornblende, biotite, augite, and quartz in the usual andesitic groundmass. Chemical analysis of the dacite shows that it contains 60 per cent of silica, or only 2 per cent more than a hornblendic andesite from the northeastern part of the district. Mineralogically and chemically the dacite at Goldfield is merely a quartz-bearing andesite and is in no way similar to the more siliceous rocks called dacite by Mr. Spurr in his report on the Tonopah district.<sup>a</sup>

The geological relation of the dacite to the andesite is not entirely clear. Its comparatively coarse porphyritic texture, the irregular outlines of its exposures (see Pl. II), and the occurrence of small bodies of dacite within larger masses of andesite are all indicative of the intrusion of the dacite into the andesite. As a rule, however, the contact between the two rocks is not well enough exposed or the rocks have been too much altered in its vicinity to afford definite proof of this relation. At one point only, situated 1 mile N. 35° E. of the summit of Preble Mountain, a sharp intrusive contact is exposed between the dacite and the andesite. The dacite is glassy at the contact and penetrates the andesite in a way that shows the latter to be the older rock. Of course this exposure merely proves that one mass of the dacite is intrusive into a certain mass of andesite. The general conclusion reached, that the dacite as a whole closely followed the andesitic eruptions and was intruded into the flows and subordinate tuffs at moderate depth, is still in need of evidence for its complete establishment. It is probable that this intrusion was accompanied

<sup>a</sup> Mr. Spurr's reasons for using the name dacite for rocks which should strictly receive other names are given in *Geology of the Tonopah mining district, Nevada*: Prof. Paper U. S. Geol. Survey No. 42, 1905, p. 59, footnote.

by some deformation of the region, and the andesite flows appear to have been somewhat eroded before the next eruptions took place.

After the dacitic intrusion a rock provisionally classed as quartz latite was erupted as a series of flows and is now extensively exposed in the low hills in the southeast corner of the district. This rock, which corresponds more closely than anything else in the Goldfield region to some of the Tonopah dacite, is in most places a porous light-gray or greenish rock with abundant glistening scales of black mica and phenocrysts of fresh plagioclase, mostly oligoclase. In some varieties quartz and orthoclase are present, and some thin flows of vitrophyric rhyolite are associated with the quartz latite. Two areas of rhyolite in the northeast corner of the district are provisionally mapped with the latite in Pl. II. The quartz latite is easily worked, and a quarry opened in this material  $1\frac{1}{2}$  miles south of Preble Mountain has afforded stone for buildings in Goldfield.

No traces of mineralization have been found in the latite, and the ore deposits of the district were probably formed prior to its eruption. This, however, is not definitely determinable, as none of the latite occurs in the immediate vicinity of known ore bodies.

After the latitic eruptions the region was covered by an extensive body of fresh water in which were laid down 500 feet or more of bedded conglomerates, tuffs, tuffaceous sands, pumiceous ash, and diatomaceous earth. These deposits, the "cement rock" of the miners, cover a large part of the district, particularly in its outskirts. They underlie the town of Goldfield and stretch both northward and southward beyond the limits of the area studied. They also extend beneath the "malapi" (malpais) mesa southwest of town and are well exposed in the steep bluffs of the ravine in which are situated the city waterworks. The beds here dip generally westward at angles ranging from  $25^{\circ}$  to  $30^{\circ}$ .

Although the lake beds appear to be locally conformable with the quartz-latite flows, yet as these are present beneath them in only a small part of the district, and as the formation of the lake indicates some change in the configuration of the region, there is at least a suggestion of erosion between the quartz-latite eruptions and the lacustrine condition. Volcanism continued active during the existence of the lake, as shown by the prevailingly volcanic nature of the sediments and the occurrence within them of a thick flow of quartz-bearing basalt and thin flows of glassy rhyolite. The quartz basalt, as shown by wells, underlies the southwestern part of Goldfield and is exposed on the automobile road to Bullfrog.

In the eastern part of the district, northeast of Preble Mountain, are some bedded breccias and tuffs from 200 to 300 feet in present thickness. They rest upon dacite and andesite and are made up largely of fragments of these rocks, particularly of dacite mingled with abun-

dant fragments of volcanic glass and some sandy tuffs. These beds are provisionally correlated with the lake beds just described.

Lacustrine conditions were ended by deformation of the region, the domical structure being accentuated or possibly initiated by this uplift. After the lake beds had been beveled by erosion and probably entirely swept away from the central part of the district eruptions of rhyolitic lavas again began.

The latest rhyolite is a light-pink or pale-brown rock, rather porous and containing abundant pumiceous fragments. It forms a flow about 100 feet in maximum thickness, which is exposed in the bluffs southwest of Goldfield, under the dark volcanic rock which caps the mesa. It is this rhyolite that is quarried for building stone at Rabbit Spring. It is in some places overlain by a few feet of unconsolidated tuffaceous sand and volcanic gravel.

Finally, the volcanic history of the region was brought to a close by the eruption of one or more flows of olivine basalt. This is the dark rock that is conspicuous as the capping of the mesa southwest of town. It also forms the summit of Myers Mountain, Blackcap Mountain, and several hills south and east of the area mapped. The larger areas of this rock represent remnants of flows which rest upon a nearly horizontal surface of older rocks; but some smaller masses, such as that shown on the east slope of Vindicator Mountain, are intrusive and may fill orifices through which the basalt reached the surface to spread out as flows.

#### CORRELATION.

While final correlation of the Goldfield rocks with those of Tonopah and other districts can not be attempted in this preliminary report, yet it may be suggested that the lake beds of the Goldfield district probably correspond to the Siebert tuff<sup>a</sup> of the Tonopah district, which Mr. Spurr regards as probably of Miocene age. If this correlation proves to be correct, the Tonopah rhyolite dacite of Mr. Spurr will correspond to the quartz latite of the Goldfield sequence. The dacite of the Goldfield district has apparently no representation at Tonopah and it does not seem possible at present to correlate the two andesitic formations at Goldfield with the "earlier andesite" and "later andesite" at Tonopah. The oldest andesite at Goldfield is petrographically unlike either of the Tonopah andesites and was succeeded by rhyolites having no representation in the northern district.

Olivine basalt occurs at one point in the Tonopah district, and as it overlies the tilted Siebert tuff it is probably of the same age as the olivine basalt of the Goldfield district. The intrusions of rhyolite represented by Mount Oddie and of "dacite" represented by Butler,

<sup>a</sup> Spurr, J. E., *Geology of the Tonopah mining district, Nevada*: Prof. Paper U. S. Geol. Survey No. 42, 1905, pp. 51-55.

Brougher, and Siebert mountains, as well as the "latest rhyolite-dacite" flow on the slopes of Oddie and Brougher mountains, appear to be later than any of the volcanic rocks in the Goldfield district.

#### GEOLOGICAL STRUCTURE.

It has already been said that the district has the general structure of a low dome, the younger formations dipping gently away from a nucleus of slightly upheaved pre-Tertiary rocks that have been exposed here and there by erosion. The concentric regularity of outcrop of the various formations about the structural center of the district, which may be considered to lie in the vicinity of Banner Mountain, is modified by two main factors. One of these is the intruded dacite; the other is a strong fault which extends from a point east of Kendall Mountain southward along the east side of Columbia Mountain to the vicinity of the Redtop mine. Here it is apparently offset by a cross fault (Pl. II), but it continues southward beyond this offset past the January mine until it disappears under the lake beds and alluvium east of Goldfield. This dislocation may be conveniently referred to as the Columbia Mountain fault.

The only body of the dacite whose structural relations can here be considered in any detail is that lying just east of the towns of Goldfield and Columbia and containing the Combination, January, Redtop, and Jumbo mines. The general relations of the boundary of this mass to the topography indicate that it is a rather irregular sheet with a gentle southwest dip. It is supposed to be intrusive, but present exposures of this particular mass afford no conclusive evidence to support this view as against the idea that it is a flow. This dacite, as indicated by the character of its southern contact and as shown by the workings of the Florence mine, extends southward under the andesite between Goldfield and Milltown. The small area of andesite shown in Pl. II between the Combination and Jumbo mines is probably an outlying remnant of the same andesitic flow that laps up over the dacite at the Florence mine.

The thickness of this dacite sheet is not known. The Jumbo shaft, sunk at a point where apparently very little of the dacite has been eroded away, was 450 feet deep at the end of 1905 and was still in this rock. Such data as are available indicate that the dacite sheet has an average dip rather under than over 10°. If the sheet was of uniform thickness prior to erosion, then the bottom of the Jumbo shaft should be very near the bottom of the dacite. It is to be hoped that sinking at this point will be continued, as the determination of the thickness of the dacite at this particular place will be of much value in forecasting the probable changes in country rock that will be experienced in the other mines with increased depth. Regularity in an intruded sheet can not, however, be assumed, and the dacite may

be much thicker in the vicinity of the principal mines east of Goldfield than is indicated by its general relation to the topography. The preparation and study of several geological structure sections, such as are planned for the final report, will probably throw additional light on the form and thickness of the dacite mass.

The Columbia Mountain fault dips to the east and has a normal throw, dacite and andesite being dropped against the older rhyolite and pre-Tertiary rocks of Columbia Mountain, as shown in Pl. II. The fault is very poorly exposed. It is followed by an inclined shaft at the Conqueror mine and is cut by the Columbia tunnel on the east slope of Columbia Mountain. Between these two points the outcrop of the fault is covered by loose material, and there are no mine workings to reveal the character of the fissure. On the south slope of Columbia Mountain the presence of the fault determines the position of a little ravine eroded in the soft, crushed material of the fault zone. The distribution of the rocks south of Columbia Mountain shows that the main fault is offset by a transverse fault having approximately the position shown in Pl. II. The rocks in this vicinity, however, are much altered and are so covered by detritus that no exposures of this fault are known. It apparently passes just southwest of the Redtop mine into an area where future mining development will probably afford satisfactory proof of its presence. South of Columbia the position of the main fault is only approximately determinable as a general line of demarcation between the locally much-altered first andesite on the west and the dacite on the east. The fault must pass only a short distance west of the January shaft, but it is not exposed at the surface. At the bottom of the January mine, however, 280 feet below the surface, a very soft zone of crushed and altered rock was encountered at the shaft. As this material was practically a mud, the drift had to be heavily timbered and lagged, and the fault zone can not therefore be fully examined. It is certain, however, that it dips to the east, that it separates dacite on the east from the oldest andesite on the west, and that it represents a zone of considerable displacement accompanied by brecciation of the adjacent rocks. It is probably the Columbia Mountain fault.

The throw of the fault is not yet determined, but its maximum is certainly over 500 feet and may prove to be of twice that magnitude. It antedated the deposition of the lake beds and is thus older than at least a part of the doming of the region.

After the lacustrine epoch the region was domed, but the elaborate faulting which followed the deposition of the Siebert tuff at Tonopah had no representation in the Goldfield region. Some faulting occurred after the eruption of the olivine basalt, as may be seen south and west of Goldfield (Pl. II), but these faults are not of great structural importance. The fact that the remnants of the olivine-basalt flows now



stand at different altitudes and occasionally slope at angles somewhat steeper than that of the broad mesa southwest of Goldfield suggests that there probably has been some differential movement or warping of the region since the basalt was erupted.

### ORE DEPOSITS.

#### GENERAL CHARACTER.

The deposits hitherto of most importance in the Goldfield district are irregular bodies of rich oxidized gold ore. These have been derived from sulphide ores, of more or less complex mineralogical character, in which pyrite is accompanied by minerals containing copper, silver, antimony, arsenic, bismuth, tellurium, and other elements. The most notable features of the oxidized ore bodies are their remarkable richness and their equally remarkable irregularity. The ores are almost without exception associated with craggy outcrops of silicified volcanic rock, although only a very small proportion of these outcrops, which are extraordinarily numerous and constitute the most striking superficial feature of the district, have been found productive. Associated with the silicification other processes of locally intense alteration have also been active, producing in many cases a softening of the rock affected and thus serving to accentuate the silicified portions under the selective action of erosion. The deposits have formed along zones of fissuring which for the most part are very irregular in trend, are rarely traceable in any one direction for more than half a mile, and are not planes of notable faulting. Branching and intersection are very common. Many of the outcrops show no linear character, being mere irregular knobs of siliceous material. Some of these probably represent silicification at the point of intersection of two or more inconspicuous approximately vertical fissures. Others are erosion remnants of nearly horizontal silicified zones and, as in the case of Black Butte, merely cap the hills whose summits they form.

#### MINING DEVELOPMENT.

The principal producing mines in the Goldfield district in the latter part of 1905 were the Combination, Florence, January, Redtop, Sandstorm, and Kendall. The Jumbo, one of the most important properties in the district, was involved in litigation at this time and had produced no ore since the early part of the year. The Quartzite mine at Black Butte, now part of the Black Butte Consolidated property, was also practically idle, although it had shortly before attained a considerable production. The Reilly lease, although on the Florence ground, was worked as a separate mine, and added about \$475,000 to the production of the district in less than two months' time.

The Combination Mines Company owns 10 claims and 3 fractional claims in the district, six of the claims forming an undeveloped out-

lying group about a mile east of the mine. At the time of visit the mine was worked through a steeply inclined shaft to a vertical depth of about 280 feet. There are 5 levels, the first a short adit 30 feet below the collar of the shaft and the others at intervals of 50 feet. The bottom level is under water. A new vertical shaft, equipped with a counterbalanced cage and a 30-horsepower electric hoist, was nearly ready for use at the end of the year. The levels run approximately northwest and southeast and the total length of ground explored is nearly 1,000 feet. On the northwest there is a connection with the January mine and on the southeast with the Reilly slope. The mine is equipped with a 20-stamp mill and cyanide plant. A tube mill was also being installed at the time of visit. Water for milling and other purposes is pumped from Alkali Spring, 10 miles northwest of the mine. The mill is driven by electricity, with steam as reserve power.

The Florence mine has a vertical shaft 350 feet deep and 6 very irregular levels. Most of the levels are at intervals of 50 feet, but between the 250-foot and 350-foot levels there is merely a short sublevel not connected with the main shaft. The general trend of the workings is about north-northwest, and the total length of exposed ground at the time of visit was, about 500 feet. Drifts were being rapidly extended northwestward, and possibly before this report appears the main workings will have been connected with the Reilly levels.

Hoisting at the Florence is done by electric power. The Reilly shaft, which lies between the main Florence shaft and the Combination shaft, is 210 feet deep, with four levels approximately 50 feet apart. These, like the Florence levels, are very irregular.

The Jumbo shaft was 450 feet deep at the time of visit and sinking was in progress. The shaft is west of the ore, and there were no cross-cuts or drifts below the 250-foot level. Above this the rich ore of the lode, which is unusually regular for this district, has been stoped by lessees through half a dozen shafts for a length of about 600 feet. A mill of two Nissen stamps was in process of building in December, 1905.

The January mine has a vertical shaft 280 feet deep, exclusive of sump, with six levels at irregular vertical intervals. All the levels are sharply curved at the shaft and run from that point about 100 feet north-northeast and 160 feet southeast, both arms of the curve running toward the Combination ground.

The Redtop mine at the time of visit was in an early stage of development, the shaft being 180 feet deep, with levels at 100 and 160 feet below the surface.

The Sandstorm mine, being worked partly by the company and partly by lessees, is rather unsystematically developed. The greatest depth attained at the time of visit was 150 feet.

The Kendall shaft is about 200 feet deep, with levels at 60 and 100 feet below the surface.

Until very recently gasoline engines of from 12 to 30 horsepower have been almost universally used in the district for hoisting and other purposes, the use of steam at the Combination and January mines being the only exception to this practice. Electric power, however, is now coming into use. The electricity is generated on Bishops Creek, 98 miles west-southwest of Goldfield, at the east base of the Sierra Nevada and is supplied by the Nevada Power, Mining and Milling Company to both Tonopah and Goldfield. The present capacity of the main line is about 4,000 horsepower. The two districts together were using about 1,500 horsepower at the close of the year 1905. The rates in force at this time for continuous service, based on peak loads measured on the motor side of transformers, were as follows:

*Rates for electric power per horsepower per month.*

	Net rate.
1 to 10 horsepower.....	\$20. 00
11 to 20 horsepower.....	15. 00
21 to 50 horsepower, \$15 less 10 per cent.....	13. 50
51 to 75 horsepower, \$15 less 15 per cent.....	12. 75
76 to 100 horsepower, \$15 less 20 per cent.....	12. 00
101 to 200 horsepower, \$15 less 25 per cent.....	11. 25
201 horsepower and upward, \$15 less 30 per cent.....	10. 50

Users of the power were required to furnish transformers and other connecting apparatus.

The present water supply of Goldfield is obtained from a well 90 feet deep, situated about three-fourths of a mile southwest of the center of town in the lake beds that underlie the volcanic capping of the mesa. The supply in September, 1905, was about 15,000 gallons a day, but experience with shallower wells at this locality has shown that the available water at any given depth tends to diminish, so that a continual supply can be secured only by occasionally deepening the well or by drifting through the water-bearing strata. The water supply of Columbia is obtained from Rabbit Spring. The general surface of the ground water of the district is not so deep as might have been expected in so arid a region. The 280-foot level of the Combination mine is under water, and the January mine has water at the same depth. This water, however, is entirely unfit for use in steam boilers. Near Diamondfield the Spokane shaft affords a good supply of water at less than 100 feet, and the Dewdrop or Highland shaft is in water below the 100-foot level. The water from the Dewdrop shaft is pumped to Columbia Mountain for use in milling. Most of the custom mills in the district derive their water from individual wells up to 200 feet in depth, sunk in the lake beds west of town.

An adequate quantity of water can probably be obtained for many years from wells and from the lower levels of the mines. Extensive and long-continued pumping operations, however, are likely to bring

to the surface more water than is supplied by precipitation, so that a slow subsidence of the general level of underground water may be expected.

#### MILLING AND SHIPMENT.

According to an estimate made by Mr. Francis A. Thomson, superintendent of the New Western Reduction Company, and published in the Goldfield Sun of September 14, 1905, the average daily production of the Goldfield mines at that time was 70 tons of shipping ore of an average value of \$200 a ton and 100 tons of milling ore at an average value of \$40 a ton. In general, ore of lower grade than \$100 a ton is milled in the district. The Combination Mines Company mills all of its ore except that exceeding \$600 a ton in value, which is shipped.

The freight rates on ore to San Francisco, Salt Lake City, or Denver in 1905 ranged from \$8 a ton for ore under \$25 in value to \$22 for \$300 ore. For richer ores, from 3 to 5 per cent of the value per ton in excess of \$300 was added to the \$22 rate. For example, on \$2,000 ore the freight would be \$22 plus 5 per cent of \$1,700, or \$107 a ton. Smelting charges were \$10 a ton on all siliceous ores, with an additional charge of \$1 a ton if the silica were unusually high.

With the exception of the Combination, none of the mines had mills in operation at the end of 1905. The other mines either shipped their ore directly or disposed of it to one of the four reduction or sampling companies in the district. Some ore of lower grade has also accumulated on the various mine dumps.

The New Western Reduction Company pays 85 per cent of the assay value for ore, subtracting a charge of \$12 a ton for milling. Improvements in this plant were in contemplation at the time of visit and these charges may since have been lowered. The Nevada Goldfields Reduction Company, popularly referred to as Frank's mill, buys ore outright and mills it or, in the case of ore from the January mine, pays 95 per cent of the assay less \$12 a ton for treatment. The American Milling and Water Company buys ore or mills it at an average charge of \$12 a ton. The Columbia Sampling and Ore Company acts as sampler or purchaser only, shipping the ore out of the district for treatment. The total maximum capacity of the three custom mills running in 1905 was about 70 tons a day. The milling facilities have probably since been increased by a new custom mill at Columbia, by the new Jumbo mill, and by improvements in the older plants. The Combination mill at the end of 1905 had just been increased to 20 stamps, with an estimated capacity of 70 tons.

#### DISTRIBUTION.

The rock alteration directly associated with ore deposition has affected all of the igneous rocks in the district that are older than the latite. The alaskite, however, shows less metamorphism than the

older rhyolites, the two andesites, and the dacite, perhaps in part because of the comparatively small areas of this rock exposed. The



FIG. 3.—Plan showing distribution of siliceous ledges east of Goldfield.

most intense and conspicuous alteration is manifest in a belt which begins in the dacite south of Columbia and just east of the Columbia Mountain fault, and stretches in a slight curve eastward over Preble Mountain and for some miles east of the area mapped. In this belt the characteristic siliceous outcrops, largely in andesite, are more abundant and more conspicuous than in any other part of the district. There is a general tendency for the more persistent fissures to conform in strike to the trend of the zone, but the departures from this direction are so numerous and varied as to leave little more than a suggestion of system. A plan of the principal outcrops of silicified rock in the western part of this belt is shown in fig. 3. The exposures in this part of the dis-

trict are better than elsewhere and probably over 90 per cent of the zones and centers of silicification are visible in outcrop. These

outcrops have been plotted with a plane table. Their representation is necessarily conventionalized, as there is usually no distinct boundary between silicified and unsilicified rock and one center of silicification may merge into another. There is no attempt in the figure to represent relative size. A circular or irregular outcrop is represented by a dot and a linear or lodelike outcrop by a line. Many small outcrops are necessarily omitted, especially where the metamorphism has been most intense and the centers of silicification are closely crowded.

A second belt of alteration extends east and west across the northern part of the district from Kendall Mountain over McMahon Ridge and Black Butte. East of Black Butte the belt widens and becomes less distinct.

Between the two belts, siliceous outcrops are scattered rather irregularly over the district, in some places, as on Banner Mountain and near the Sandstorm mine, being clustered into local groups of some prominence or importance.

The distribution of ore is more restricted than that of the siliceous outcrops. The most important ore bodies thus far found are in dacite and are all within a small area, less than three-fourths mile square, lying just east of Goldfield and Columbia. The ore bodies of the Combination, Florence, Jumbo, January, and Redtop mines are all in this limited portion of the dacite mass, and those of the St. Ives, Simmerone, and Commonwealth mines occur farther east in the same rock. The rich ore shoots of the Sandstorm and Kendall mines are in the oldest effusive rhyolite. No important deposits have yet been found in the oldest andesite, although the occasional finding of small bunches of ore has encouraged prospecting in this rock, particularly in the area south of Columbia. The only large body of ore thus far opened in the younger andesite is that of the Quartzite mine at Black Butte. Most of the smaller properties north of Diamondfield are, however, in this andesite, which is also the country rock of the Gold Bar mine, 1 mile northeast of Myers Mountain.

No cause has been found for the preponderance of ore in the dacite. The two andesites have undergone similar alteration and do not differ greatly in petrographic character from the dacite. It is probable that the dacite is lithologically no more suitable for ore deposition than the andesites and older rhyolites, and future prospecting may considerably decrease the relative economic importance of this apparently more favored rock.

As the latite, lake beds, youngest rhyolite, and basalts show no evidence of siliceous alteration or of ore deposition, they are probably younger than the primary ores. This conclusion, however, is less definite than it might be had not these younger volcanic formations been eroded completely away from what appear to be the areas of most intense alteration.

## FORM OF DEPOSITS.

There is no name in the literature of economic geology that is strictly applicable to the deposits of the Goldfield district. They can not properly be called veins, since in their formation fissure filling has had a wholly subordinate, in fact, almost negligible, part. In many cases they are not even approximately tabular in form and are not genetically related to any single main fissure. Walls are generally lacking except such slips as may have occurred since the original ore deposition.

In general, the deposits are irregular masses of dense flinty quartz formed by the silicification of fractured or shattered rock of various kinds by silica-bearing solutions. In describing them it will be necessary to distinguish between these masses as a whole and the bodies of ore that may or may not be associated with them. As the silicified masses form characteristic rough outcrops they may be

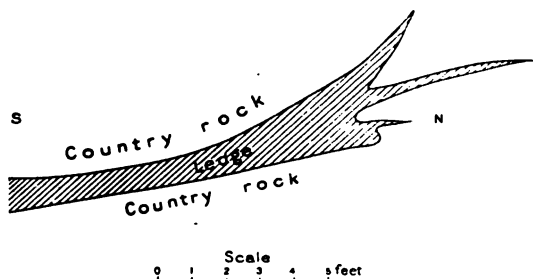


FIG. 4.—Vertical section through an irregular flat ledge as seen in the west wall of the Black Butte tunnel.

conveniently referred to as *ledges*, ledge being a word in common use among miners for any outcropping vein, lode, or less regular deposit, while the ore bodies proper will be designated *pay shoots*. The pay shoots, almost without exception, occur

in or near the ledges, but there are hundreds of ledges which, so far as known at present, do not contain a pound of ore.

In plan, particularly as shown in outcrop, the ledges may be roughly circular, crescentic, rectilinear, or wholly irregular. Some of the circular or irregular masses are approximately horizontal remnants capping small knobs. Black Butte furnishes a good example of this type, a tunnel run through the hill having shown conclusively that the great silicified mass forming the summit of the butte is merely superficial and that similar but thinner zones of silicification traverse the kaolinized andesite beneath it. A section of one of these minor ledges as exposed in the wall of the tunnel is shown in fig. 4, the mode of branching and pinching being thoroughly characteristic of these deposits. Others may, as Mr. Spurr suggests,<sup>a</sup> have the form of pipes or chimneys, although mining operations have not yet demonstrated the descent of pipelike masses to any great depth. Many of the irregular ledges, such as miners would ordinarily term blow-outs, are situated at the intersections of lines of fissuring and silicification.

<sup>a</sup> Bull. U. S. Geol. Survey No. 260, 1905, p. 134.

The most important ledges, those upon which most work has been done and which have produced the greater part of the ore, are more or less linear or lodelike in plan. Notwithstanding the great irregu-

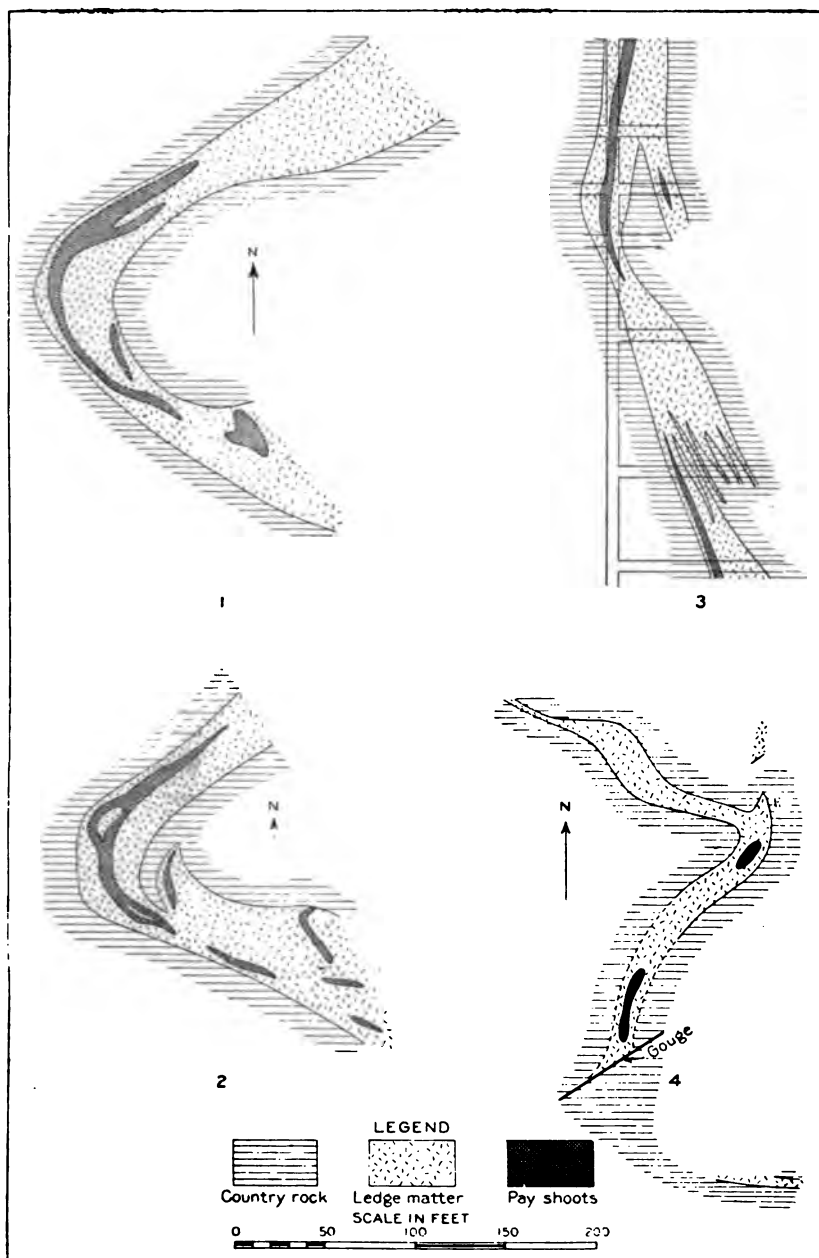


FIG. 5.—Ledges and pay shoots in the Goldfield district: 1, Plan of pay shoots and ledge on the first level of the January mine; 2, plan of pay shoots and ledge on the second level of the January mine; 3 diagrammatic cross section of the January ledge and pay shoots, looking north; 4, plan of pay shoots and ledge on the second level of the Florence mine.



larity of their associated pay shoots, the ledges of the Sandstorm, Kendall, Redtop, January, Combination, Jumbo, and Florence mines, as well as those of many less prominent properties, have this general lodelike character. The Sandstorm-Kendall ledge can be followed over the surface as a continuous outcrop of intensely silicified rhyolite for a distance of fully

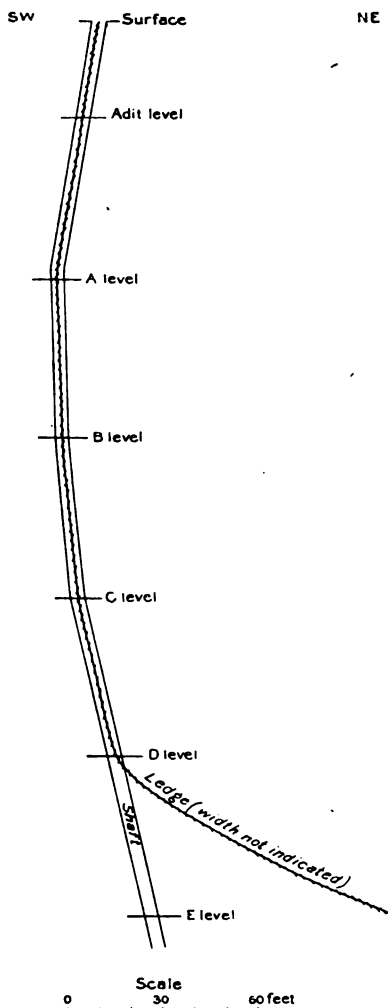


FIG. 6.—Cross section through the Combination mine, showing change in dip of the Combination ledge.

they do the soft unsilicified country rock. Consequently, within the zone of oxidation there is usually a remarkably sharp change in color and texture from the rusty siliceous material of the ledge to the gray, soft, pyritized country rock, locally referred to as porphyry. The lack of definiteness of some of the ledges at a depth of a few hundred

1,500 feet. The Combination, Reilly, and Florence workings are all situated on a ledge of silicified dacite and andesite, which, although not quite continuous at the surface and somewhat curved, apparently represents one general zone of fissuring nearly half a mile in length. The Jumbo ledge has a fairly regular linear outcrop for a distance of 600 feet. The January ledge is sharply curved in plan, and the Redtop, owing to its width and the shortness of its outcrop, is less lodelike than the ledges of the other mines mentioned. Inspection of fig. 3 will show that a linear outcrop is not peculiar to the ledges of known economic importance, but is characteristic of many whose value is as yet unproved. The width of the ledges is variable, as may be seen from fig. 5, ranging in most cases from 10 to 100 feet.

As none of the ledges has yet been explored below a depth of 400 feet, less is known of them in vertical section than in plan. Moreover, below the zone of oxidation the distinction between ledge and country rock in some cases becomes very indefinite. Oxidizing solutions, as a rule, penetrate the brittle, shattered ledge matter more readily than

feet is not entirely a matter of oxidation, however. In some cases silicification unquestionably decreases gradually with increase in depth.

Changes in dip are common. The Combination ledge has a dip of  $81^{\circ}$  to the southwest down to a depth of 80 feet. Below that it dips northeastward. At a depth of 230 feet the ledge, as shown in fig. 6, becomes very flat and exhibits little silicification. Unfortunately this part of the ledge, being under water, could not be seen at the time of visit. The Reilly ledge, which is continuous with what is known as the "No. 2 shoot" of the Combination mine, dips to the northeast for the first 160 feet below the surface, but then turns and dips in the other direction, as illustrated in fig. 7. The roll in the ledge is not regular or horizontal, as is shown by the fact that the levels of the Reilly workings, seen superposed in plan, are not parallel, but cross one another at considerable angles. This irregularity is the more remarkable, as the "No. 2 vein," in the Combination mine, contains one of the most regular pay shoots in the district. Similar changes of dip are very pronounced features of the ledge exploited in the main Florence workings. Although most of the levels are only 50 feet apart, a general plan of the mine shows them crossing and recrossing one another at angles of  $30^{\circ}$  or more, indicating abrupt changes in dip and strike.

In general, all the underground work done in the district shows that the ledges, although they have some lodelike features, are subject to such remarkable changes in dip and strike that methods of mining based on the behavior of ordinary veins and lodes are not applicable to them. Such eccentricities, as are shown in figs. 4 to 7, illustrate the uncertainties attendant upon underground exploitation and indicate that any attempt to carry development work far ahead of the known ore becomes largely a game of chance.

If the ledges are irregular, the pay shoots are even more so. In most cases these occur within and are completely inclosed by ledge matter; but there are some exceptions to this rule and some ore bodies

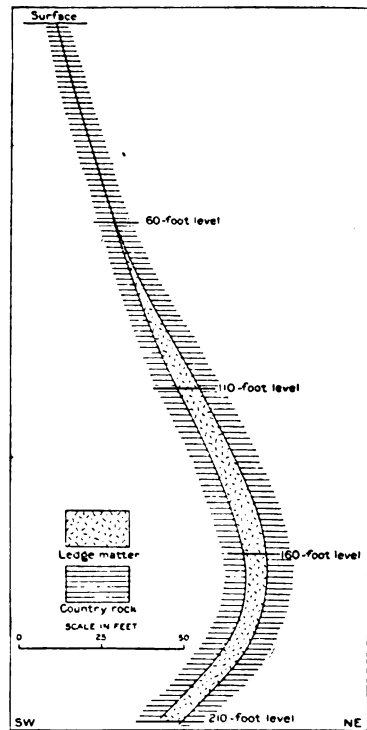


FIG. 7.—Cross section of the Reilly ledge, showing changes in dip.

are directly in contact with unsilicified country rock. Some of the pay shoots are mere bunches or "pockets." On the Velvet claim, for example, several thousand dollars' worth of rich ore was found at the surface, but efforts to find a continuation of this shoot in depth have thus far been futile. The St. Ives, Simmerone, and Conqueror (Tonopah Club) mines have had similar histories, although this in itself does not warrant the conclusion that deeper ore bodies may not yet be found in these properties. The Kendall mine at the time of visit had produced about \$100,000, all from ore within 60 feet of the surface, and the deeper workings at that time showed no ore worth shipping. The rich ore of the Sandstorm mine has all come from very irregular pay shoots, which in many cases rest on flat floors of hard, barren, silicified

rhyolite. These bodies are usually found at the intersection of later cross fissures with the main silicified ledge. Although some rich ore has recently been opened at a depth of 100 feet, the bulk of the production has come from ore within 50 feet of the surface. The ore bodies first worked in the Combination mine occurred in short irregular shoots, most of them at points of intersection of two or more fissure zones and with a tendency to a vertically elongated or pipelike form. A sketch plan of one of these shoots on level A is shown in fig. 8, and in fig. 9 are represented plans of shoots on the level below. At greater depth these irregular pay shoots appear to come together, and on level D the deposit is lodelike,

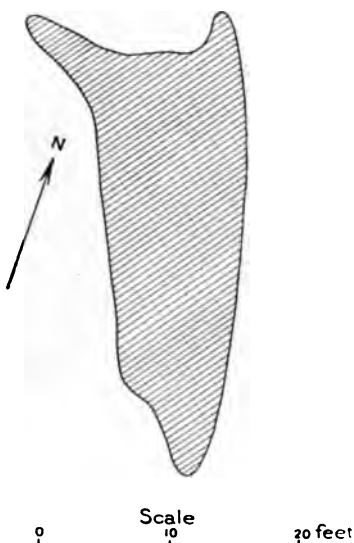


FIG. 8.—Sketch plan of pay shoot on A level of the Combination mine.

with a length of about 250 feet. About 100 feet of this is shipping ore, the rest being of milling grade.

The so-called "No. 2 shoot" of the Combination mine is, with the possible exception of the Jumbo pay shoot, the longest and most regular body of ore known in the district, having been stoped on the 130-foot or B level (fig. 9) for over 300 feet. Moreover, it is separated by only a short, barren interval from the Reilly ore body, which is on the same line of fissuring. This pay shoot is the hanging-wall portion of a shattered silicified ledge, 30 to 40 feet wide, which strikes northwest and dips northeast at angles ranging from  $75^{\circ}$  to  $80^{\circ}$ . The ore, which is up to 6 feet in width, has no walls and can not always be distinguished by the eye from the barren or low-grade ledge matter. It lies near, but not always in contact

with, a persistent gouge, which separates the oxidized silicified ledge matter from soft kaolinized and pyritized dacite. Although this pay shoot is so regular and persistent on the B level it apparently extends to the surface at only one point as the pipelike shoot of unusually rich ore which was followed in the No. 3 prospect shaft. Below this level the relations of the No. 2 pay shoot to the original Combination pay shoot are yet to be determined. As shown in fig. 9, the two shoots probably come together, although, as already pointed out, no constancy of direction can be safely assumed in this district, even for so apparently regular a ledge and pay shoot as the No. 2 of the Combination mine, especially as the same ledge in the Reilly workings is subject to remarkable changes in dip. In fact, attempts to find the No. 2 shoot on the D level of the Combination mine have thus far been unsuccessful.

In the January mine the erratic distribution of irregular pay shoots within the curved ledge is well shown in fig. 5. The two levels chosen for illustration are only 50 feet apart, yet the distribution and outlines of the ore bodies are notably different. The distribution of pay shoots in the Florence mine (fig. 5) is also without recognizable plan. The main ore body, from which the lessees obtained most of their ore, lies mainly between the 200-

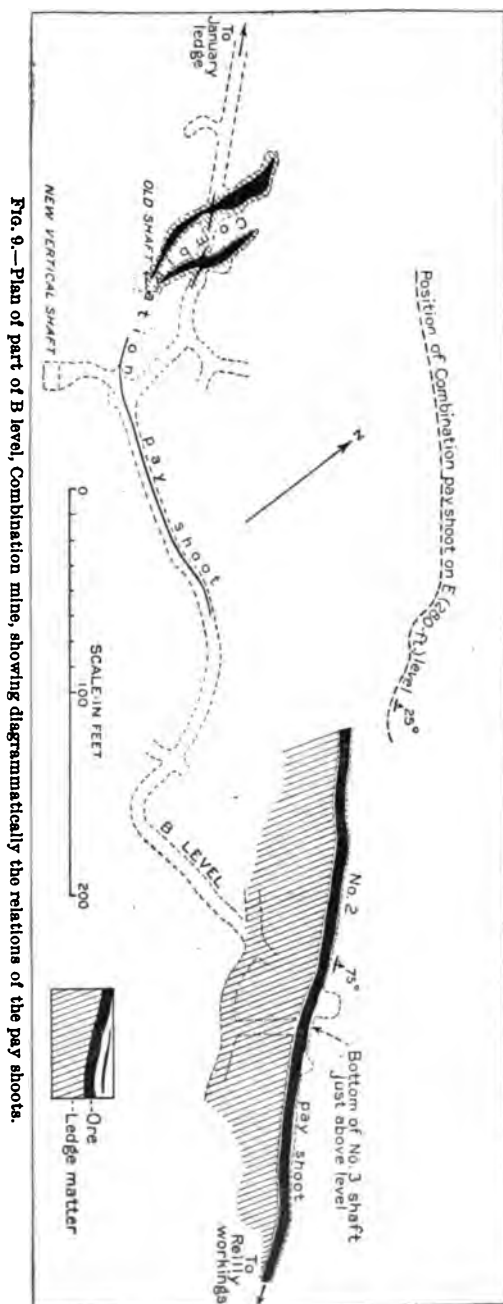


FIG. 9.—Plan of part of B level, Combination mine, showing diagrammatically the relations of the pay shoots.

foot and 150-foot levels, and the stope opened in it is the largest in the district. This pay shoot is a rudely lenticular mass dipping from  $35^{\circ}$  to  $40^{\circ}$  to the southwest. The stope is fully 75 feet long and up to 18 feet wide. The ore continues for a short distance below the 200-foot level, steepening to a dip of  $70^{\circ}$ . It consists of much shattered dacite (or possibly andesite), partly silicified, full of fine pyrite and traversed by countless seams of kaolin or similar soft white material. It grades into ledge matter without any definite walls.

#### MINERALOGY.

The unoxidized ore of the district, as exemplified in the Combination, Florence, Jumbo, and January mines, consists of pyrite, bismuthinite, and a reddish-gray copper mineral with the general composition of tetrahedrite. Native gold is usually associated with these minerals and in the rich ore may be easily visible. These minerals are at some places arranged in successive layers or crusts and in others are irregularly dispersed through a siliceous gangue, which usually shows evidence of being shattered and silicified rock.

The brown copper-bearing mineral contains antimony and sulphur and will be provisionally referred to as tetrahedrite. The absence of crystal form, however, makes it impossible to make a definite determination, as some rarer minerals, such as famatinite, have a chemical composition similar to that of tetrahedrite but differ in crystallization. Whether the tetrahedrite from Goldfield contains notable quantities of silver or gold has not yet been determined. It is usually an indication of rich ore.

Bismuthinite, the sulphide of bismuth, occurs as slender lead-gray prisms and needles and is regarded by the miners as a certain sign of good ore.

The richest unoxidized ore of the Combination, January, and Florence (including the Reilly) workings consists of free gold, usually in particles so fine that the auriferous portions of the ore look, at first glance, as if they were stained with ocher. The gold is intimately associated with tetrahedrite and bismuthinite. Pyrite is usually present also, but there is apparently no relation between the abundance of this mineral and the richness of the ore.

Concentric shells of ore minerals about silicified rock fragments are rather characteristic of the rich ore. Specimens from the Combination mine show an inner zone of free gold and quartz, up to an eighth of an inch in thickness. This is covered by a shell of tetrahedrite and this by an outer crust of radial iron sulphide, probably pyrite and not marcasite. In the Florence mine similar fragments show, first, a shell of pyrite, then one of tetrahedrite, and, finally, a thick crust of quartz speckled with native gold, tetrahedrite, and pyrite, and trans-fixed by needles of bismuthinite. Some of the particles of gold are

embedded in the compact quartz, others are inclosed in the bismuthinite. The different crusts are not in every case sharply defined nor are they necessarily continuous.

Tellurides occur sparingly in the Goldfield ores. A so-called telluride ore from the Reilly lease was found by Dr. W. T. Schaller, of the chemical laboratory of the Survey, to contain only a trace of tellurium. A dark-gray mineral occurring in small specks in compact white quartz and silicified andesite, in the Jumbo Extension mine near Diamondfield, is a telluride of gold, but the mineral is not sufficiently well crystallized for satisfactory determination of its species. As Dr. Schaller reports the absence of silver, the telluride can hardly be petzite. A similar mineral from the Goldfield-Belmont mine, three-fourths mile north of Diamondfield, contains gold and tellurium, but no silver or lead. The material, however, is not sufficiently abundant or well enough crystallized to permit specific identification. Tellurium has also been detected by Dr. W. F. Hillebrand in ore collected by Mr. Spurr<sup>a</sup> from the Combination and January mines.

A small speck of sphalerite was found by Mr. W. H. Blackburn in the Goldfield-Belmont mine, but galena has not been noted in any of the mines so far as known. Chalcopyrite, a common mineral in most mining districts, was not found at Goldfield in the course of the present investigation, but it is probably not entirely absent, and is said to occur sparingly in the Sandstorm and Florence mines.

The common gangue of the unoxidized ore is quartz. This is usually compact, almost flinty in texture, and in most cases bears unmistakable evidence of having resulted from the silicification of dacite, rhyolite, or andesite. Large vugs and conspicuously crystalline quartz, such as are found in typical veins all over the world, are practically absent from the Goldfield district, where the free development of quartz crystals in open spaces is represented only by drusy films lining pores left by the solution of phenocrysts or incrusting small interstitial cavities in brecciated material.

Associated with the quartz in much of the ore are soft white substances, such as are usually termed "talc" by the miners. In some cases the white material is kaolinite (hydrous silicate of aluminum), in others it is sericite (silicate of aluminum and potassium), but one interesting result of the examination of the so-called "talc" is that a large part of it is composed of the mineral alunite, a hydrous sulphate of aluminum and potassium. Gypsum, while not known with actual sulphide ore, does occur crystallized with quartz and pyrite below the zone of oxidation in the Goldfield-Belmont mine.

No sulphantimonites or sulpharsenites of silver were noted in the mines at the time of visit. A specimen of rich ore from the Florence mine, in the possession of Mr. F. O. Altinger, an assayer in Goldfield,

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<sup>a</sup> Ores of Goldfield, Nev.: Bull. U. S. Geol. Survey No. 260, 1905, pp. 137 and 138.

showed, however, a small speck of ruby silver (proustite) and some tabular crystals which were apparently polybasite or stephanite.

Cinnabar is distributed rather abundantly through a mass of intensely altered andesite about 2 miles east of Black Butte in crystalline particles and aggregates up to an inch in diameter. The deposit has not been developed, but appears to be at least 150 feet long and 50 feet wide, so that it may prove to be of commercial importance. Native sulphur occurs in a similar manner, but in bunches up to 6 inches in diameter, in a bleached andesitic breccia about a mile east of Tognoni Springs. Small quantities of sulphur have also been found in the croppings of the Blue Bull mine. It is doubtful whether the mineral is abundant enough to be of economic importance.

Oxidized ores have supplied a large part of the gold produced, and in some mines, particularly the Sandstorm and Kendall, no sulphide ores are as yet known. As a rule the oxidized ore is a soft, shattered, more or less earthy material, usually stained brown by oxide of iron. Fragments of rusty porous quartz are mingled with kaolin, alunite, gypsum, alum, oxide of iron, and various earthy mixtures of no definite mineral composition. Some of the rich ore, as in the Sandstorm mine, is a nearly white, impure kaolin, gritty from the presence of minute crystals or grains of quartz and containing all through it abundant specks of free gold. Barite, in crystals up to an inch in length, is abundant in this ore. In the Redtop mine some of the richest ore is a firm gritty ocher, of bright yellow color, containing abundant very fine particles of gold. In January and Combination mines some of the most characteristic rich ore consists of porous rusty quartz (silicified dacite), in which the pores and crevices are partly filled with yellow, earthy limonite and tiny pearly scales of bismite, the oxide of bismuth. In some specimens these scales form pseudomorphs after bismuthinite. They are usually a sign of rich ore, but are occasionally found in low-grade or barren material. Native alum, or kalinite, and alunite are rather abundant in the oxidized ore of the January mine. In the mines at Black Butte the richest oxidized ore usually shows little greenish-yellow specks of a ferric tellurite, which Dr. W. F. Hillebrand has determined to be either emmonsite or durdenite. The presence of this mineral shows that the original sulphide ore contained one or more tellurides. Tellurite, a honey-yellow crystalline mineral, which is the oxide of tellurium and should not be confused with the salts of tellurous acid known to chemists as tellurites, was noted in a partly oxidized telluride ore from the Goldfield-Belmont mine near Black Butte. Melanterite (hydrous ferrous sulphate), in some cases mixed with a very little chalcantite (hydrous copper sulphate), occurs in the partly oxidized ore of the Combination and Florence mines.

The valuable constituent of the oxidized ores is native gold. Native silver or halogen compounds of silver have not, so far as known, been

recognized in the Goldfield district. The gold, which has undoubtedly undergone some concentration in the process of oxidation, is in most of the ores very finely divided, and even in the richest ores might easily be overlooked by one not accustomed to its appearance. Some of the Sandstorm ore on close examination resembles a fine, somewhat rusty, sandstone having gold as the cementing material.

#### GENESIS OF THE ORES.

Much office and laboratory work remains to be done before the genesis of the Goldfield deposits can be satisfactorily discussed. A few suggestions bearing upon this problem are all that can be offered at this time.

The formation of the ledges was initiated by extensive fissuring of all the rocks of the region with the probable exception of the latite and younger formations. The fissures formed a complex network, in which there is no obvious system, and although the disturbance was of a kind to produce broad zones of cracked and even shattered rock it did not in general dislocate the formations by persistent or structurally important faults. This network of irregular fissures furnished channels more or less devious to hot ascending waters carrying silica, hydrogen sulphide, or alkali sulphides, and possibly sulphurous or sulphuric acid. The waters held in solution also gold, silver, bismuth, copper, antimony, arsenic, and other metals, as well as small quantities of tellurium. A large part of the pyrite associated with the ores was undoubtedly formed by the action of sulphur-bearing solutions on the ferruginous silicates of the country rock, but some of the iron was probably brought up in solution also. The effect of these waters was to convert large masses of the fissured rocks to quartz—rhyolite, andesite, and dacite all being altered by molecular replacement to siliceous masses very similar in general appearance and in places where the alteration was most intense retaining but faint traces of original igneous texture. Other portions of the same rocks were changed to soft aggregates of kaolin, sericite, alunite, and pyrite, the two kinds of alteration apparently going on side by side. The practical absence of carbonates from the ore deposits is noteworthy.

It has been a common practice in the district to refer to the silicified portions as dikes of rhyolite and to the soft, pyritized country rock as porphyry. This is misleading, as ledge and country rock were in all cases originally the same eruptive rock.

The ledges are complex structures. The first silicification seems in no instance to have produced the ore as mined to-day, but was followed by at least one other period of fissuring and brecciation, as is shown by the fact that the rich sulphide ore often coats fragments that are themselves veined and silicified.

How far this rich ore was deposited by ascending solutions and how far by solutions that percolated downward through a zone of oxidation



is a question still under investigation. It is probable that to a large extent the ores were the result of deposition from rising solutions.

Another question of great practical importance relates to the changes in the ore with increase of depth. There is considerable evidence in favor of the conclusion that ores of the grade now required for shipment are not likely to extend to as great a depth as 1,000 feet. Available workings certainly show a tendency of the sulphide ores to decrease in value below a depth of 300 feet. It should be said, however, that the mines as a whole are too shallow to establish this tentative conclusion as a fact.

#### VALUE OF THE ORES.

As a whole the ores thus far mined at Goldfield are unusually rich. Prior to January 1, 1905, the Combination mine shipped a total of 1,166 tons of ore having an average assay value of \$419 per ton. This ore averaged 20.22 ounces of gold and 2.68 ounces of silver per ton. The best ore from the mine was taken from the small No. 3 shaft, which afforded about 309 tons valued at \$159,000, or \$514 per ton. Approximately 59 tons of this averaged, according to Supt. Edgar A. Collins, 54.8 ounces of gold per ton, which is equivalent to a value of nearly \$1,100. This ore, which was oxidized, contained only 1 ounce of silver to each 20 ounces of gold.

Mr. J. E. Spurr<sup>a</sup> estimated in 1904 that the average value of the ores mined in the Goldfield district was from \$200 to \$300 per ton or more. In 1905 the average value of shipping ore was about \$200 per ton and of milling ore from \$40 to \$50 per ton.

Shipments averaging over \$1,000 per ton are by no means uncommon. One of 14½ tons from the Kendall mine is said by Mr. Spurr<sup>a</sup> to have yielded \$45,783 net in the preliminary milling, while the tailings still contained about \$1,000 a ton. A shipment of sulphide ore from the Florence mine, made in 1905, just before the date of visit, averaged 100.8 ounces of gold, 3.5 ounces of silver, and 3.5 per cent of copper, giving a gross value of over \$2,000 per ton. The silver in this shipment was unusually high for an ore from this part of the district. An analysis of a sample of Florence ore published in the Goldfield News of April 7, 1905, is as follows:

#### *Analysis of sample of Florence ore.*

Gold.....	ounces per ton..	70.21
Silver.....	do.....	7.8
Copper.....	per cent..	6.9
Iron.....	do.....	28.5
Zinc.....	do.....	.3
Arsenic.....	do.....	.3
Sulphur.....	do.....	13.20
Silica.....	do.....	43.44

<sup>a</sup> Loc. cit., p. 133.

This analysis is evidently neither accurate nor complete. If, as is probable, the copper was present as tetrahedrite, there should be considerable arsenic or antimony in the ore. The figures cited, however, in connection with those previously given, serve to indicate that the silver is largely combined with the copper in the mineral tetrahedrite.

Some extremely rich ore was obtained from the Reilly lease, occasional specimens several inches in diameter being nearly one-half gold. Several shipments from this lease are said to have assayed \$20,000 a ton, and, according to Mr. Frank Oliver, one lot of over 4 tons was worth from \$6,000 to \$7,000 a ton.

Some of the ore from the vicinity of Diamondfield carries a greater proportion of silver than that from the mines near Goldfield. A specimen containing tetrahedrite, taken from the Quartzite mine of Black Butte, yielded on assay 11.06 ounces of gold and 115.70 ounces of silver per ton. Samples of ore with tetrahedrite from the Goldfield-Belmont mine assay, according to Mr. W. H. Blackburn, yielded from 1 to 2 ounces of gold and from 30 to 60 ounces of silver per ton. A similar high proportion of silver is found in the ore of the Jumbo Extension mine. Some bunches of ore in the Blue Bull mine also show a very high proportion of silver, one assay giving three hundred and eighty-two times as much silver as gold.<sup>a</sup>

Although, as is to be expected in a new district, much of the very rich ore has been oxidized, yet some of the sulphides in the mines near Goldfield have exceeded in value anything found in the oxidized zone above them.

#### MINE MAPS.

Very few of the mines at Goldfield have satisfactory maps of their underground workings, and it is consequently in many cases difficult to understand the relation of one ore body to another in the same ledge or even to ascertain the shape and dimensions of individual pay shoots. This condition will doubtless be improved as the mines become older and as work is systematized, but the present development work in the larger mines could undoubtedly be laid out to better advantage if good plans of the levels were supplemented by numerous accurate sections and if important fissures, ledge matter, and ore were plotted upon the mine maps. Models constructed of sheets of glass, such as are used by the largest mines at Cripple Creek, would be especially useful as aids to understanding the relations of the ore bodies to one another in space, and if kept up to date and intelligently studied would doubtless furnish suggestions for exploration. Where ore bodies are so irregular in shape and distribution good maps and practical models are necessities rather than luxuries and cost less than many a crosscut driven at random into barren ground.

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<sup>a</sup> Spurr, J. E., loc. cit., p. 137 (footnote).

## BULLFROG DISTRICT.

### INTRODUCTION.

In the autumn of 1905 a topographic map of the most important part of the Bullfrog district, covering an area about 7 miles from east to west and  $2\frac{1}{2}$  miles from north to south, was made by Mr. William Stranahan, topographer, on the scale of 1:24,000, with contour intervals of 20 feet. This map shows the town of Beatty near its eastern margin and just includes the Original Bullfrog mine on the west. Geological work was begun in December of the same year and completed in March, 1906. Messrs. W. H. Emmons and G. H. Garrey mapped the geological formations and studied in detail the relations and character of the rocks, while the writer devoted a shorter time to a general survey of the field and to the examination of the ore deposits.

The present account of the district is merely a preliminary sketch, in advance of a complete report, now in preparation.

### SITUATION OF THE DISTRICT.

The name "Bullfrog district" is usually rather vaguely applied to a large tract of desert hills and mountains extending far to the north and west of the area particularly investigated. For convenience of description, however, the name as used in this report will refer in most cases to the area mapped, which includes nearly all the ore deposits thus far developed, although there are some scattered prospects of considerable promise lying without its boundaries.

The position of the district is shown in the index map (fig. 1) on page 9. It lies 60 miles south-southeast of Goldfield, with which it is connected by stage and automobile lines from 70 to 80 miles in length and about 20 miles northeast of the great trough of Death Valley. Railroads are in course of construction into the district from the south, one from Las Vegas on the San Pedro, Los Angeles and Salt Lake Railroad, and one from Ludlow, Cal., on the Atchison, Topeka and Santa Fe Railway, by way of Death Valley. Railway connection is projected, also, between the Bullfrog and Goldfield districts.

The principal town is Rhyolite, situated at the south base of a short east-west range of hills that connects the Funeral or Grapevine Mountains on the west with Bare Mountain and other irregular groups of peaks, ridges, and mesas on the east, and separates the Amargosa Desert on the south from a similar desert basin extending northward past Stonewall Mountain toward Goldfield.

The other towns in the district are Bullfrog, which adjoins Rhyolite on the south, and Beatty, 4 miles east of Rhyolite, on the so-called Amargosa River, a feeble trickle of water that is fed by springs a few miles north of the town and is finally absorbed in the sand and gravel of the Amargosa Desert.

## HISTORY.

Long prior to the discoveries of ore at Tonopah and Goldfield the springs in Oasis Valley, just north of the site of Beatty, proved attractive to a few desert wanderers who took Indian wives and established rude ranches along the Amargosa River, the only stream of drinkable water within a radius of 40 miles. Prospectors traveling northeastward from Death Valley or northwestward from the springs at Ash Meadow would pass the night at one of these ranches, perhaps spend a few days examining the hills in the neighborhood, and then again brave heat and thirst on the shadeless plains.

The remarkable development of Tonopah and the finding of rich ore at Goldfield stimulated prospecting throughout all of southwestern Nevada, and the rapid advance of the material adjuncts to civilization into this most inhospitable region greatly lessened the hardship and danger that had hitherto attended a traveler far from bases of supply and vitally dependent upon springs, often long distances apart and not always readily found. In the autumn of 1904 the Bullfrog claim was located on a prominent outcrop of quartz, about 3 miles west of the site of Rhyolite. The name, which afterwards became that of the district, is said to have been suggested by the green color of the ore. The ironical humor of associating a paludinous fauna with scorching aridity is characteristic of the Nevada pioneers.

The usual rush of prospectors followed the first discovery, and most of the ground between the Bullfrog claim and the Amargosa River was soon covered with claims. A settlement of tents, known as Amargosa City, sprang up near the Bullfrog claim, but as the center of interest shifted eastward to Bonanza and Ladd mountains the first town was abandoned for the settlements of Bonanza, Bullfrog, and Beatty. In February, 1905, a town-site company laid out the streets of Rhyolite and, by offering a certain number of lots free, succeeded in a few days in establishing this as the chief town of the district. Whether it will remain so in spite of the inducements offered by other town-site companies and the proposal to carry the main railway lines past Beatty remains to be seen. It has, however, an advantage of position with regard to the mines that is much in its favor.

## PRODUCTION.

Work in the Bullfrog district up to the end of the year 1905 had been confined mainly to prospecting and to blocking out ore. The Montgomery-Shoshone mine is said to have shipped about 100 tons of rich ore, the original Bullfrog about 13 tons, and the Denver at the beginning of the year 1906 had shipped 1,000 sacks. As all ore at the time of visit had to be hauled by wagons to Goldfield or Las Vegas, the general tendency among the mine owners was to delay shipping until the entrance of a railroad should afford better facilities.

## TOPOGRAPHY.

The Bullfrog Hills attain a maximum elevation of 6,095 feet above sea, the highest point being about a mile beyond the northern boundary of the area covered by the detailed map. The hills, which are steep, rocky, and practically bare of vegetation rise sharply from the gently sloping desert plains that border them on the north and south. The marginal elevation of these plains ranges from about 3,300 feet on the Amargosa River at Beatty to about 4,500 feet at Mud Spring 4 miles north of Rhyolite, and the local relief of the hills is thus about 2,800 feet. Southeast of Beatty, Bare Mountain, which culminates in a peak 6,235 feet high, presents a steep southwest front to the Amargosa Desert and an eastern face almost as precipitous to an embayment of the same desert. On the north, however, the mountain, which is composed principally of schist, limestone, and quartzite, merges into hills which, in spite of the topographic break due to Oasis Valley, are structurally and lithologically an eastern continuation of the Bullfrog Hills. To the west, the Bullfrog Hills extend without any definite topographic or geological break into the Grapevine Mountains.

The hills in the area particularly described in this report constitute a very irregular group, partly buried by the thick alluvium characteristic of the broad, undrained basins of arid Nevada. The upper limit of the alluvium, unlike that at Goldfield, is remarkably definite, and above it the rocky structure of the hills is laid bare to a degree highly satisfactory to the geologist. A view over the district from any high point suggests at once that the panorama spread out before the observer owes its main features to the cooperation of faulting and erosion on a thick series of lavas. Many of the ridges are obviously fault blocks, most of them tilted to the east, so that their western scarps show the horizontal banding of successive flows while their northeast faces exhibit merely the irregular pinnacles and ravines characteristic of the erosion of a dip slope of a single rock layer of generally homogeneous texture. Moreover, as one follows with his eye along one of the ridges the individual white, gray, green, pink, brown, or black bands that represent the edges of different flows, he finds that they can never be traced far before they are sharply offset or cut off entirely in a manner that can be due only to faulting. Thus it is evident that the faults are not parallel but intersect one another, and the irregularity of the relief, the absence of continuous lines of parallel ridges and valleys, is seen to be the visible expression of a complexly faulted structure. How far this expression is direct and how far it has been modified or transformed by erosion will be considered in another place. There are no perennial streams in the district except the Amargosa River, but erosion, though fitful in its

activity, proceeds at times with great energy, and the water that falls upon the hills undoubtedly accomplishes more erosive work than would the same quantity under the conditions prevailing in a humid country well covered with vegetation. The arid, stony slopes of the Bullfrog Hills are elaborately carved by running water, and the so-called "box canyon" a mile west of Rhyolite is striking testimony to the power of a stream that is probably active for only a few hours or a few days in the year.

## GENERAL GEOLOGY.

### INTRODUCTION.

Although the writer has personally supervised the general geological work in the district and has himself thoroughly traversed the area, most of the facts in the following brief description of the general geology are derived from the notes of Messrs. Emmons and Garrey, to whom credit should be given for the actual working out of the intricate faulting and for the discrimination and correlation of the various lithological units. Their complete geological map and a more thorough discussion of the geology than is here possible will form parts of the final report on the district, which is now in preparation. They are not directly responsible for this preliminary outline, some of the statements in which may require modification as the material gathered in the field is more thoroughly studied.

### THE ROCKS.

The prevailing rocks of the area studied—the ones that give character to the topography and that contain the ore deposits—are rhyolitic flows, presumably of Tertiary age. Much older rocks, however, are exposed in the southwestern and southeastern parts of the district and these will first be briefly described.

Three miles west-southwest of Rhyolite, south of the stage road (Pl. III), is a little group of low, rounded, whitish hills, composed of schistose and gneissic rocks cut by numerous irregular dikes of pegmatite. Some of the schists are practically quartz schist. Others are quartz-muscovite schists, and there are a few bands of amphibole schist. At one place there is a small exposure of dolomitic limestone, which appears to be an integral part of the crystalline formation, although it is not notably metamorphosed. It is possibly a small residual of a limestone bed that once covered the schistose rocks. Intimately associated with the schists are bands of biotitic gneiss with drawn-out, eyelike feldspars. The pegmatite dikes are very numerous and very irregular. In most of them quartz is more abundant than feldspar, and no essential difference could be detected between the white vitreous quartz that forms part of pegmatite dikes and the

similar quartz that, without the accompaniment of feldspar, forms bunches and irregular lenticular veins in the gneiss and schist. Both

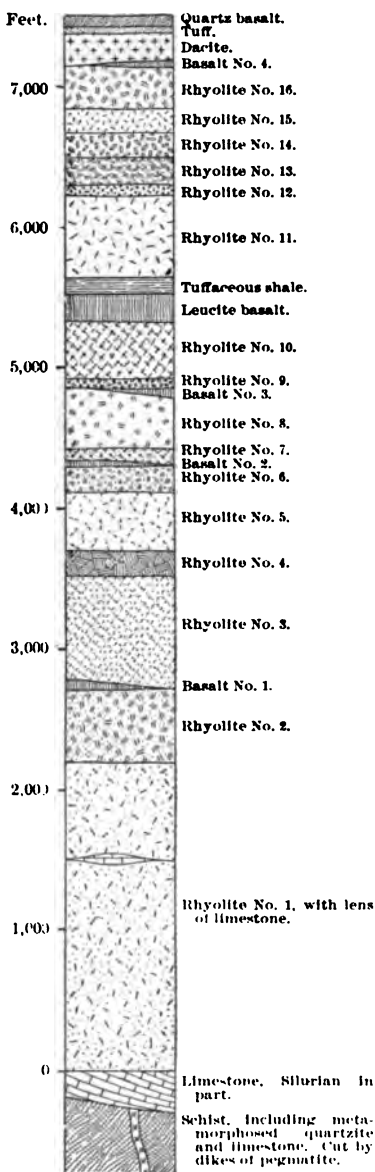


FIG. 10. —Generalized columnar section of the rocks of the Bullfrog district.

places, however, the cleavage crosses the bedding planes. The limestone is recrystallized and where originally it graded into calcareous

carry a little pyrite. The metamorphic rocks are cut by a dike of rhyolite, probably once connected with one of the flows to the north, and by two varieties of dark hornblending porphyry.

North of the stage road and a short distance west of the Original Bullfrog mine, the schists are overlain by gray limestone. Fossils collected by Mr. Garrey from a similar limestone about a mile west of the area mapped are regarded by Mr. E. O. Ulrich as of Silurian age. The schists are thus probably pre-Silurian.

For the distance of nearly a mile east of the Original Bullfrog mine the rocks are concealed by alluvium, but on the eastern edge of this alluvial embayment a little of the schist and limestone is exposed. East of this exposure the alluvium of the Amargosa Desert laps up over the Tertiary volcanic rocks.

About 4 miles east of Rhyolite and due south of Beatty, schistose rocks are again exposed between the alluvium and the rhyolite, forming relatively low hills in the southeast corner of the district and extending eastward into Bare Mountain, which affords magnificent sections of their structure. These rocks are clearly metamorphosed limestones, shales, and quartzites. The schistosity is generally parallel with the beds, which are bent into gentle folds and somewhat faulted. In a few

shales it now shows a gradual passage from crystalline limestone through calcite schist into mica schist. The quartzite has in part been changed to quartz schist. The schists are cut by a few pegmatite dikes, but these are not nearly so abundant as in the southwest corner of the district. At one place bunchy lenticular quartz veins in the schist are cut by a pegmatite dike. Veins of white quartz are fairly abundant, but are short and irregular. Some of them carry a little galena, but no other sulphide was noted.

In Bare Mountain the schists are apparently overlain by thick light-gray limestones, which are not particularly metamorphosed and are probably younger than the thin-bedded yellow limestones in the schists. Whether the schists are of the same age as those west of Rhyolite is an open question, although such general evidence as is available seems to favor an affirmative answer.

The total thickness of the rhyolite flows, including a few relatively thin layers of basalt, is estimated at from 7,000 to 8,000 feet. The provisional columnar section (fig. 10) will give some idea of their sequence and relative thicknesses. As shown in this section, 16 different rhyolitic formations are distinguished and will be represented on the final geological map. These formations are not all individual flows. Some probably consist of several superposed sheets of rhyolite of similar lithological character. Others may be portions of flows which by reason of pronounced difference in color or texture are conspicuous elements in the landscape and are important aids in the investigation of the faulting which is the principal feature of the local structure. It is obvious that for the purposes of description and reference each of the rhyolitic units must receive some distinctive designation. The usual practice is to apply to each formation the name of the place where it is most typically developed or exposed, as, for example, the "Beatty rhyolite;" but even were there sufficient geographic names available, their application to so large a number of geological units of the same general kind of rock would heavily burden the reader's memory and would probably lead to confusion. It seems best, therefore, to number the various rhyolitic formations from the base upward. Four flows of feldspathic olivine basalt will be similarly distinguished from one another. Should detailed work in adjacent areas require the interpellation of additional flows between those here recognized, they might if it is desired to retain the numbering now proposed, be designated "rhyolite No. 2 a," "rhyolite No. 13 a," and so on. It will probably be long, however, before any such expedient will be necessary. The estimates of thickness indicated in the columnar section are rough approximations and are likely to be somewhat modified in the final report.



The basal member of the Tertiary volcanic series, rhyolite No. 1, is best exposed in and near the deep ravine locally known as the "box canyon," about  $1\frac{1}{4}$  miles west of Rhyolite. It is here much cracked and jointed and weathers in rather rounded yellowish masses. Rhyolite No. 1 undoubtedly includes more than one flow and shows considerable variation in texture. Much of it is fine grained and slightly porphyritic, often showing abundant small spherulites in the groundmass. Such spherulitic varieties rarely contain phenocrysts of quartz. In places the rock grades into aphanitic glassy rhyolites in which the laminae due to flowage are so thin and regular that slightly weathered exposures have a remarkably close resemblance to outcrops of shales. Other facies are regularly banded flow breccias which in places simulate coarse sandstones or grits. At one point, about  $2\frac{1}{4}$  miles northwest of Rhyolite, a lens of dark-gray limestone occurs in rhyolite No. 1, showing that this formation is not a single flow, but represents at least two eruptions separated by an interval within which limestone to a thickness of several feet was deposited in a probably local basin.

In addition to the large area in the vicinity of the "box canyon," rhyolite No. 1 is exposed on the western edge of the district near the Original Bullfrog mine and on the east side of the Amargosa River at Beatty. The maximum thickness of this formation is provisionally estimated at 2,200 feet.

Rhyolite No. 2, which directly overlies rhyolite No. 1, is a flow breccia, usually light gray, but in some places of dull purple or pinkish color, and consists of a glassy groundmass, generally showing lines of flowage, in which are embedded fragments of rhyolite and basalt. The fragments are generally less than 2 inches in diameter and in most cases are darker than the matrix. This rhyolite is well exposed on the hill just north of the Original Bullfrog mine and on the slope above Buck Spring. Its thickness is estimated at 500 feet.

Between rhyolites No. 2 and No. 3 a flow of basalt intervenes at a few places, particularly near Buck Spring, 2 miles northwest of Rhyolite, where the flow attains a maximum thickness of 40 feet. The rock is an ordinary olivine-bearing feldspar basalt and may conveniently be referred to as feldspar basalt No. 1.

Rhyolite No. 3, which forms the greater part of the hill between Buck and Sullivan springs, 2 miles northwest of Rhyolite, resembles in its lower part the flow breccia of rhyolite No. 2, but grades upward into a somewhat vesicular glassy rock with pronounced flow lines. Its thickness varies from 300 to 500 feet. None of this rock is exposed in the eastern half of the district, although it probably occurs below the present surface.

Rhyolite No. 4 is variable in color, the common tints being greenish-white, pale bluish-gray, pink, dull purple, light buff, and cream. It

is of porous texture and rarely shows conspicuous crystals. This flow is about 200 feet thick in Bullfrog Mountain (U. S. land monument 170), north of the Original Bullfrog mine. It is exposed also on the north edge of the district northeast of Buck Spring, but is not known east of this point.

Rhyolite No. 5 is a characteristic and important member of the volcanic sequence. It forms the summit of Bullfrog Mountain, where it is approximately 500 feet thick. The west slope of Bonanza Mountain is made up of this rock, as is also the lower western slope of Ladd Mountain, just east of Rhyolite. The same rhyolite is the principal rock of the southern and eastern slopes of Sutherland Mountain, of which Bonanza Mountain is a southeastern spur. It is thus the country rock of the Denver mine.

The principal lithological characteristics of this formation are the presence of phenocrysts of black mica and the frequent occurrence of spherulites, some of which, on Sawtooth Mountain, are over 18 inches in diameter and others east of Beatty attain a diameter of 5 feet. The lower part of the formation is in most places a well-defined band of brown spherulitic or perlitic glass up to 50 feet in thickness, and a similar band occurs at some localities about 200 feet above the base of the formation, which has a total thickness of from 500 to 800 feet. In the vicinity of the Denver mine the rock is compact, shows very few porphyritic crystals, and is prevailingly buff in tint. Farther west, however, this variety grades into rather more typical facies.

Rhyolite No. 6 is in most places a nearly white, porous, somewhat pumiceous flow breccia, with occasional pale-greenish or pinkish tints. It is this formation that makes the prominent white band visible from Rhyolite on the slopes of Busch Peak and of Ladd and Sutherland mountains. It is also exposed along the western bases of many of the hills between Ladd Mountain and the Amargosa River. The thickness of rhyolite No. 6 varies from a few feet in Bullfrog Mountain to about 300 feet on the southwest slope of Montgomery Mountain.

One notable feature of rhyolite No. 6 is the occurrence within it of masses of basalt. Some of these are irregular. Others are thin tabular bodies which stand nearly vertical and which, did they occur alone, might easily be mistaken for dikes. Close examination of the contacts, however, shows that the rhyolite was fluid after the basalt had solidified. Little tongues of rhyolite penetrate the darker rock. The flow banding of the rhyolite, moreover, conforms locally to the surfaces of the basalt masses. That the apparent dikes are really inclusions is certain, but no satisfactory explanation has yet been found for their vertical attitude in a flow that must have had a generally horizontal movement or for the source of the basaltic material. These inclusions are best seen near the trail from Rhyolite to Sullivan Spring, where it passes through the saddle in the ridge south of Busch Peak.

East of Rhyolite the white No. 6 rhyolite is directly overlain by a very persistent and characteristic pink rhyolite, which is No. 7 of the rhyolitic sequence. In the ridge west of the town, however, a basaltic flow, feldspar basalt No. 2, intervenes between the two rhyolites. This basalt is well exposed on the trail from Rhyolite to the Eclipse and Denver mines, and forms a conspicuous black band around the upper part of Sutherland Mountain. It is vesicular in some parts, and the base of the overlying rhyolite is in most places crowded with basaltic fragments apparently picked up from the scoriaceous upper surface of the flow.

The pink rhyolite forming the seventh of the lithological divisions of the rhyolitic series is exposed at many places throughout the district. It is particularly conspicuous in the west face of Ladd Mountain and in the corresponding slopes of the various hills between Ladd Mountain and Beatty. Its maximum thickness is probably about 60 feet. By reason of its characteristic color and the wide distribution of its exposures it has proved a very useful formation in the study of the faulting undergone by the district, and in Pl. III is distinguished from the other formations in order to illustrate in part the general structure.

Rhyolite No. 8 is the most widespread and extensively exposed of the many rhyolitic formations in the district. It forms the eastern slope of Bullfrog Mountain, the main part of Busch Peak, the summits of Sutherland and Bonanza mountains, and the summit and east slope of Ladd Mountain, and occurs in most of the short ridges between Montgomery Mountain and the hills of pre-Tertiary rocks south of Beatty. This rhyolite is generally dark gray but in many places, as on Busch Peak, has a dull purple tint and, like most of the siliceous members of the volcanic series, exhibits considerable color variation. Quartz phenocrysts are abundant and the matrix being usually rather compact the rhyolite appears to resist erosion better than most of the flows. The lower 60 feet of the formation, whose maximum thickness appears to be about 600 feet, is in some places a volcanic glass.

Rhyolite No. 9 is a gray or nearly white, rather pumiceous flow breccia varying from 25 to 150 feet in thickness. It rests in most places directly upon rhyolite No. 8, but in the hills just north of Rhyolite and on the northeast slope of Ladd Mountain a flow of basalt (feldspar basalt No. 3) intervenes. This flow breccia is eroded with comparative ease and is seldom conspicuous, occurring for the most part in saddles or on the smooth slopes in the eastern half of the district.

Rhyolite No. 10 is a brownish-gray flow breccia containing fragments of rhyolite, basalt, and, rarely, schist. The matrix contains abundant phenocrysts of quartz and clear feldspar. The base of

the formation is in many places a rhyolitic glass. The formation is extensively exposed in the eastern half of the district. It occurs in the hills due north of Rhyolite, being in most cases the summit rock, and forms the summits and most of the eastern slopes of Montgomery, Paradise, and Velvet mountains. Its greatest thickness is probably over 400 feet.

Overlying this rhyolite on the northeast side of Velvet, Paradise, and Montgomery mountains, and probably also in the valley west of Rainbow Mountain, is the thickest flow of basaltic rock known in the district. Its maximum thickness may be provisionally estimated at 200 feet. In the field this rock was not supposed to be essentially different from the other basaltic flows in the district. Microscopical study shows, however, that it is a fresh, typical leucite basalt, a type of rock that is not common and that apparently has never been recorded in Nevada. The specimen upon which the present determination rests was taken from an exposure five-eighths of a mile west of Black Peak or about  $1\frac{1}{2}$  miles north of Rhyolite. The dark basaltic rock crossed at various points on the road from the Montgomery-Shoshone mine to Beatty is probably of the same type, and a small mass, three-fourths of a mile south of Beatty, is known to be leucite basalt.

The leucite basalt is overlain between Beatty and the Montgomery-Shoshone mine by thin-bedded shales, some of which show worm casts, and by other fine-grained sediments. The total thickness of these beds is probably under 200 feet. They appear to be composed mainly of glassy volcanic detritus and to represent a brief inter-volcanic period of sedimentation, possibly in a small basin.

Above these sediments lies rhyolite No. 11, a series of flow breccias which near the base show a very close resemblance to tuffs or mud flows. Indeed, it is not always possible in the field to decide whether a given layer flowed as a molten mass crowded with fragments or as a hot pasty mud. The thickness of this formation is 500 or 600 feet.

Rhyolite No. 11 and the remaining members of the local geological section are all exposed on the west slope of Rainbow Mountain and on Black Peak.

Rhyolites Nos. 12 and 14 are both perlitic glasses, not unlike dark-green bottle glass in appearance. The upper, more prominent band is perhaps 200 feet in thickness. They are separated by rhyolite No. 13, which is also very glassy but is brown instead of green.

Rhyolites 15 and 16 are both nearly white flow breccias, distinguished mainly by the fact that bunches of dense volcanic glass or obsidian are much more abundant in 16 than in 15. No. 16 was the last of the rhyolitic outflows so far as any record has been preserved in this district. It was followed, without any recognized erosion interval, by a thin flow of basalt, which apparently did not entirely

cover the rhyolite. Resting partly on the basalt (No. 4 of the feldspar basalts) and partly on rhyolite No. 16 is the dacite flow which caps Black Peak, whose name was evidently suggested by the somber contrast of the dacite to the light and brightly tinted rhyolitic bands of Rainbow Mountain.

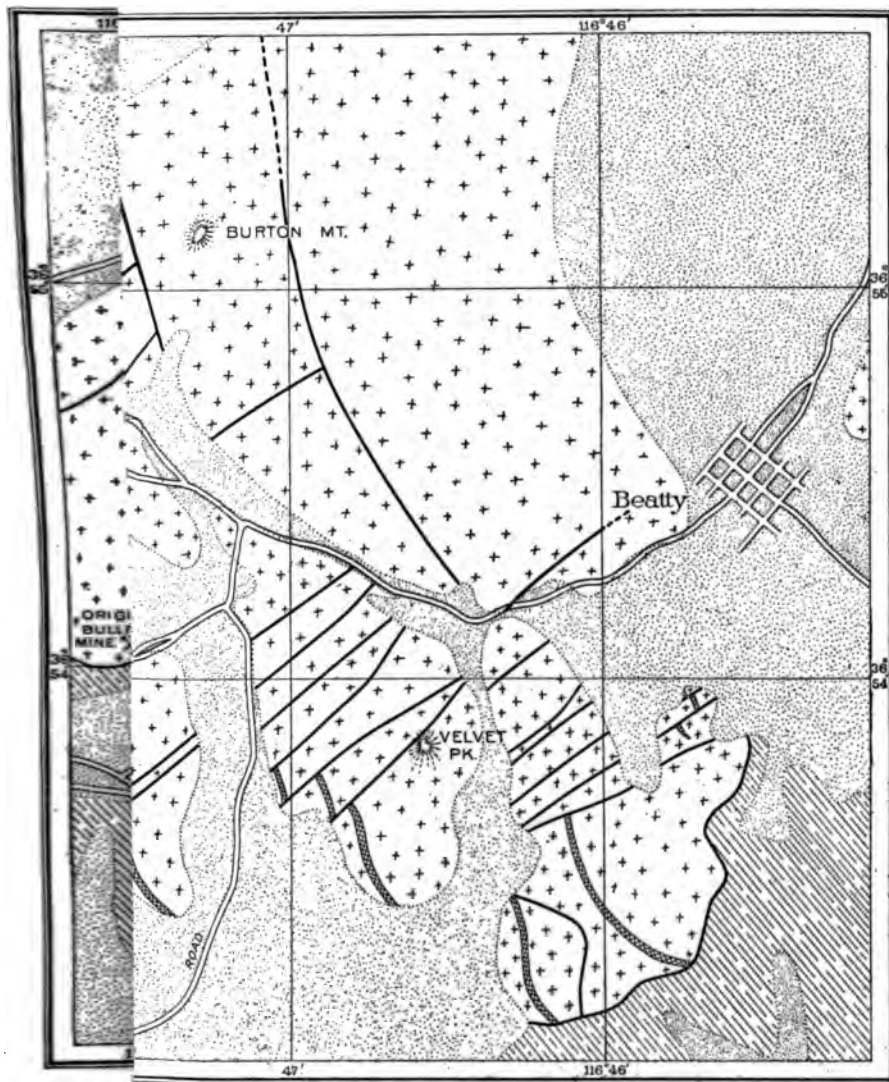
Above the dacite, and exposed in the saddle east of the summit of Black Peak, lie some thin beds of sandy tuff, probably not over 20 feet in thickness. These are overlain by a flow of quartz-bearing basalt, the youngest effusive rock in the district. In the district studied the dacite and quartz basalt occur only on the eastern slope of Black Peak. There is reason to think, however, that they have a greater distribution to the north.

No large bodies of intrusive rock occur in the Bullfrog district, but dark, basaltic dikes, usually narrow, are fairly abundant. Some are ordinary olivine basalts, but no microscopic study of the dike rocks has yet been made, and it is possible that leucite or nepheline may occur in some of them. These dikes cut the rhyolitic flows and, although perhaps not all of the same age, they are on the whole so closely related to the faulting as to indicate that they are the latest products of volcanic action preserved in the region. Some of their peculiarities will be described in connection with the structural features of the district in the following section.

The youngest formation in the district is the gravelly alluvium or wash, which, as may be seen from Pl. III, covers a large part of the area. The superficial part of this deposit is composed mainly of angular or imperfectly rounded rock fragments derived from the neighboring hills. Little is known, however, of the character of its basal portion, as the conditions under which the material accumulates are not favorable to the development of deep gorges, such as would furnish natural sections. A shaft has been sunk in the alluvium, about three-fourths of a mile south of the summit of Ladd Mountain, to a reported depth of 330 feet. The dump, which contains some rounded boulders a foot or more in diameter, indicates that the bottom of the shaft had not reached solid rock when work was stopped.

#### STRUCTURE.

At the close of the Tertiary rhyolitic eruptions the schists, gneisses, quartzites, and limestones forming the ancient platform upon which the lavas were extravasated had been buried beneath approximately 8,000 feet of nearly horizontal volcanic flows. Over the area of the Bullfrog district these flows appear to have been fairly uniform in thickness. How far beyond this they once extended is not yet known, nor has it been definitely determined whether Bare Mountain, composed of pre-Tertiary rocks, owes its elevation entirely to postvolcanic



Alluvium



ment or whether it originally stood above the general surface which the lavas spread. In the one case the volcanic rocks have stripped from it by erosion. In the other it may never have been completely buried. The former suggestion is regarded as the more probable.

After the volcanic outflows ceased the rocks were subjected to stresses which were finally relieved by faulting. There is no evidence that the volcanic formations were folded. They appear to have been broken by numerous fissures into many blocks, which were individually displaced by the dislocations. The main fissures trend from north and north to northwest and southeast. They dip to the west in most cases, and the fault blocks are tilted to the east. The faulting is thus normal in character, and one of its most striking results is the repetition across the whole district, of blocks containing the same assemblages of eastward-dipping formations, as may be seen in Pl. III.

In addition to the dominant faults just referred to there is another system characterized by nearly northeast-southwest trends. In general the throws of the northeast faults are less than those of the north to northwest faults, but a few northeast faults show profound displacement. Both systems of faults are represented in Pl. III, but the structural importance of the north to northwest faults is somewhat masked by the facts that they are in some cases partly or wholly covered by alluvium and are less numerous than those of the other system. The structure shows that under the band of alluvium between Bullfrog Mountain and the "box canyon" must lie concealed the most important fault in the district. Another important dislocation must be covered by the alluvium between Ladd and Montgomery mountains. Most of the faults are nearly vertical, but there are apparently two notable exceptions to this rule. The contact between the rhyolites and the pre-Tertiary rocks in the vicinity of the Original Bullfrog Mountain dips to the north at an angle of  $18^{\circ}$  to  $20^{\circ}$ . It is not well exposed, but seems to be a plane of some disturbance. As the various igneous formations of Bullfrog Mountain dip to the east at considerable angles and are successively cut off at this contact, they must formerly have flowed against a steep slope of Paleozoic rocks or else have been dropped by a fault. The latter seems on the whole more probable, although it is difficult to understand how normal faulting could take place on a plane so slightly inclined unless the displacement had a large horizontal component.

North of Beatty the relation of the Tertiary volcanic rocks to the Tertiary metamorphic rocks is similar to that just described. The contact is probably a fault, although the evidence for this conclusion is not as satisfactory as could be desired. East of Beatty, however, the rhyolites are clearly faulted down against the limestones and quartzites of Bare Mountain. The fault here dips northward at



angles between  $50^{\circ}$  and  $65^{\circ}$  and the rocks adjacent to the fissure are more or less crushed.

In general, the rhyolites of the Bullfrog Hills appear to be bounded on the south by a nearly east-west fault or fault zone which has dropped them against a pre-Tertiary metamorphic terrane forming the mass of Bare Mountain and exposed in a few low hills along the northern edge of the Amargosa Desert. The entire downthrown mass has been divided by other faults into numerous small blocks.

A large number of the faults are accompanied by basaltic dikes. In some cases the dike fills the fault fissure and is not itself disturbed. In other cases the faulting is in part later than the intrusion, so that the dike has been fractured or crushed by the movement. A very common feature of these dikes is their lack of continuity, the basalt occurring here and there along a persistent fissure. This peculiarity is probably in part due to movements subsequent to the intrusion, but along many of the fissures the basalt appears to have been intruded only at intervals.

#### ORE DEPOSITS.

##### MINING DEVELOPMENT.

The Montgomery-Shoshone, situated about  $1\frac{1}{2}$  miles northeast of Rhyolite on the road to Beatty, is the best known mine in the district, on account of both the size and richness of its blocked-out ore body and the legal contests that have been waged for its possession. Negotiations, which afterwards terminated in the sale of the mine, were entered into during the geological study of the district, and were unfortunately considered by the former owners a sufficient reason for withdrawing permission to enter the underground workings. The occurrence of the Montgomery-Shoshone ore has therefore not been carefully studied, although Messrs. Emmons and Garrey, who entered the mine before permission was withdrawn made some preliminary notes, which have been utilized in the present report. The main Montgomery-Shoshone shaft was 150 feet deep at the time of visit, with three levels. The Polaris mine adjoins the Montgomery-Shoshone on the west, the Polaris shaft being about 700 feet southwest of the Montgomery-Shoshone shaft. Northeast of the latter is the Providence shaft, 100 feet deep. The Steinway shaft, about 2,300 feet west of the Montgomery-Shoshone, and the Yankee Girl, in the flat just northeast of Rhyolite, are prospects which at the time of visit were about 200 feet deep. On the south slope of Montgomery Mountain are the Rand and Crystal shafts, 100 feet deep in January, 1906, but not in ore.

On the west slope of Ladd Mountain the most important workings are those of the Bullfrog National Bank Company, a shaft 200

feet deep with two levels, and of the Bullfrog Mining Company, a tunnel that was about 550 feet long on January 19, 1906.

On the steep south slope of Bonanza Mountain is the Gibraltar mine, opened by three tunnels a few hundred feet in length. The upper tunnel (No. 3) is on what is known as No. 4 vein, the one below it (No. 2) is partly a crosscut and partly on the same vein, while the lowest (No. 1) tunnel is a crosscut. No. 1 tunnel enters the east slope of Bonanza only a few feet above the alluvium, No. 2 tunnel is about 85 feet higher, and No. 3 about 60 feet above No. 2.

North of the Gibraltar, and lying mostly on the west slope of the mountain, are the properties of the Eclipse Mining Company and two claims, known as the Tramp No. 1 and Tramp Extension, which are under nearly the same ownership as the Eclipse group. The Eclipse ground is opened by the Eclipse shaft, 125 feet deep; the Hobo incline, 155 feet deep; the Lester shaft, 60 feet deep, and the Tiger tunnel, which was between 300 and 400 feet long at the time of visit. This tunnel enters Bonanza Mountain near its western base and is intended to crosscut the Eclipse and Lester veins below the bottoms of existing shafts. The Eclipse and Tramp groups are still in an early stage of development, no stoping and very little drifting having been done.

About 1,500 feet northwest of the Eclipse shaft is the Denver mine, opened by three tunnels separated by vertical intervals of 70 to 80 feet. A fourth tunnel has been started 100 feet below No. 3. The tunnels in general follow the vein, none of them at the time of the visit being over 300 feet in length.

The principal workings of the Original Bullfrog mine comprise a tunnel with several hundred feet of branching drifts and crosscuts and two shafts, one of which is 140 feet in depth.

The Gold Bar, about 4 miles northwest of Rhyolite, lies outside of the area covered by the detailed map of the Bullfrog district. The main shaft is 150 feet deep, and there are several hundred feet of drifts on two tunnels. Another promising outlying prospect is the Mayflower, situated about 7 miles north of Rhyolite and opened by a shaft which at the time of visit was 100 feet deep. The Happy Hooligan mine, 9 miles a little south of west from the town of Rhyolite and developed by a tunnel and a 40- or 50-foot inclined winze, presents some interesting geological features, which will be referred to later.

There are many other prospects in the district, some of which may ultimately prove more important than one or two of those named. Most of them, however, have been very little developed as yet or are not in ore, and it is not necessary to enumerate them in a brief report of this character.

## GENERAL CHARACTER OF THE DEPOSITS.

Attention has already been drawn to the elaborate network of faults that constitutes so important a structural feature of the district. The ore deposits are for the most part nearly vertical mineralized faults or fault zones in rhyolite. Of the many faults shown in Pl. III comparatively few have proved ore-bearing, although it must be remembered that the district is young and that a considerable number of the faults discovered in the course of the geological mapping have not yet attracted the attention of prospectors. The presence or absence of mineralization bears no evident relation to the particular variety of rhyolite constituting the country rock or to the amount of dislocation along the fissures, but appears to be mainly a matter of locality. In certain restricted areas, such as Bonanza Mountain, several fissures will show more or less mineralization, while elsewhere the same and other fissures are barren. As none of the mines has yet gone below the zone of oxidation, direct observation of the original character of the lodes is nowhere possible. All accessible portions of them have been more or less altered by the oxidation of the sulphides, by the solution of some of the gangue constituents, and by later movements along the fissures.

Most of the lodes instead of being simple veins are fissure zones containing numerous stringers of vein material and in most cases showing no definite walls. The principal stringers are parallel with the sides of the lode as a whole, but they are linked by numerous irregular cross veinlets, and similar small stringers extend for varying distances into the country rock. The lodes range in width from a few inches to 10 or even 100 feet. They are in many places accompanied or followed by well-defined regular planes or walls that have resulted from movement along the fissure zone after the original formation of the vein. Such a secondary wall may be seen in the Eclipse and Tramp shafts on the Eclipse vein.

Originally the stringers consisted of quartz and calcite carrying finely disseminated auriferous pyrite. The calcite, possibly associated in some places with other carbonates, varies in abundance. It forms a large part of the Hobo and Louisville veins on Bonanza Mountain, while it is nearly or quite absent from the National Bank vein on Ladd Mountain. The larger stringers, such as those of the Hobo vein, at many places exhibit regular depositional banding or crustification. Many of the stringers are distinct fissure fillings, with a definite contact between them and their rhyolite walls. Cases of transition from vein filling to country rock, due to the silicification of the latter, are by no means lacking, however, although this process has not been carried to anything like the extent observable at Goldfield. Much of the quartz, including some good ore, has a fine granular texture and has evidently formed by the silicification of shattered or crushed rhyolite. Typical

vein quartz, such as is characteristic of the gold veins of the Appalachians or Sierra Nevada or such as is found in the pre-Tertiary schists of the Bullfrog district, does not occur in the mines near Rhyolite, with the exception of the Original Bullfrog. The quartz is pre-vaillingly fine grained, often of a porcelainlike texture, and is usually intercrystallized with calcite.

The presence of so much calcite in veins traversing rhyolites, rocks exceptionally low in lime, is not yet fully accounted for. It is known, however, that a considerable part of the pre-Tertiary terrane upon which the volcanic rocks were poured out consists of limestone. There is a suggestion, therefore, that the vein-forming solutions gathered the calcareous constituent of the gangue from these underlying limestones.

In the process of oxidation, which in most of the lodes has been facilitated by movements that have fissured or shattered the original filling, the crystals of pyrite are changed to specks of limonite within which may occasionally be seen particles of native gold. The calcite is partly dissolved and partly changed to fragile cellular pseudomorphs of quartz. In this alteration the silica attacks first the outside of the calcite grain or crystal and works inward along cleavage planes. The final result is a shell of quartz divided by thin quartz septa or partitions, parallel groups of which are in many cases inclined to one another at the characteristic angles of the cleavage rhombohedron of calcite. Between the septa there is usually more or less black or brown earthy material, in part oxide of manganese. The development of earthy hydrous oxide of manganese is very characteristic of the oxidation of the calcitic veins of the district, and large portions of the lodes are made up of soft, dark, manganiferous earth associated with residual masses of the original quartz and calcite and containing vugs and druses of secondary deposits of these two minerals.

The ore bodies of the Montgomery-Shoshone and Original Bullfrog mines are both somewhat exceptional. The principal ore body of the Montgomery-Shoshone is a great, irregular mass of soft kaolinized rhyolite at the junction of a series of nearly north-south fissures with the basalt dike that occupies the Montgomery-Shoshone fault. The Original Bullfrog deposit is a large, nearly horizontal body of coarsely crystalline and chalcedonic quartz which lies in the fault zone between the rhyolite and the Paleozoic limestone.

The association of some of the ore bodies with basaltic dikes raises the question whether the latter have in any way influenced ore deposition. Field study shows that the dikes were intruded prior to the introduction of the gold-bearing solutions and indicates they are related to mineralization only through the fact that they occupied some of the fissures which were subsequently reopened sufficiently to permit ore deposition. There is no reason to suppose that the ores

are genetically connected with the basaltic intrusions and it is by no means certain that eruptions of dacite and rhyolite did not continue after the last basaltic dike had been injected.

#### THE ORES.

All the ore thus far mined or opened up is more or less oxidized and in most cases contains no sulphides. In the Original Bullfrog mine there is a little chalcocite or copper glance, and in some undeveloped veins in the schists south of Beatty there are specks of galena, but the only sulphide thus far found in the other deposits is pyrite. Native gold, alloyed with various proportions of silver, is the only valuable constituent of most of the ores, although cerargyrite or horn silver is fairly abundant in the rich ore of the Montgomery-Shoshone mine and more detailed study may reveal the presence of some of the halogen compounds of silver in the other deposits. The gold is finely divided, and is almost invariably found in the quartz, not in the calcite. Its characteristic association with little limonitic specks, representing oxidized pyrite, has already been referred to. In the rather exceptional Montgomery-Shoshone ore the gold and cerargyrite occur in a soft, impure kaolin formed by alteration of rhyolite. The Original Bullfrog ore consists of quartz carrying free gold, chalcocite, chrysocolla, malachite, and azurite. The quartz occurs in great variety of crystallization and color. Coarsely crystalline radial masses and banded chalcedonic forms are both common, and the colors range from clear white or pale amethyst to translucent yellow, brown, or green. In some parts of the vein calcite is crystallized with the quartz.

The ores, as sacked, range from \$100 to \$700 a ton, but the deposits on the whole are very much lower in grade than those at Goldfield. It is evident that the future prosperity of the camp must depend upon the successful working of ore much inferior in grade to that sacked for shipment under present conditions. Just what will be the lower limit in value of profitable ore and what is likely to be the general average value of ore for the whole camp under the more favorable conditions expected to follow the completion of a railroad into the district can not yet be determined.

The proportion of silver varies greatly in different mines even when these are close together. In the Denver ore, for example, the silver is said to be almost negligible, whereas the pale gold or electrum from the Gibraltar mine may contain equal parts of gold and silver by weight, although the average proportion of silver is considerably less. In the ore from the Montgomery-Shoshone mine, according to Mr. Ralph I. Johnson, assayer, the average ratio of gold to silver in 160 samples was 1 ounce of gold to 25.44 ounces of silver. This agrees fairly well with the statement made at the mine that about 45 per cent of the value in the \$700 ore is in silver.

## NOTES ON INDIVIDUAL MINES.

## MONTGOMERY-SHOSHONE.

The Montgomery-Shoshone workings are in rhyolite on the southeast side of a basalt dike. The dike occupies a structurally very important fault which dips northwestward. As a result of this dislocation the rocks exposed on Rainbow Mountain and Black Peak, northwest of the fault, which belong to the upper part of the volcanic series, are brought into juxtaposition with the older flows making up Montgomery Mountain, on the southeast side of the fault. The throw of the fault has not yet been carefully calculated, but provisional estimates show that it probably exceeds 2,000 feet. The summits

of Black Peak and Montgomery Mountain both lie on the general line of strike of the eastward-dipping flows, but while Black Peak is capped with quartz basalt (see Pl. III), Montgomery Mountain is capped by rhyolite No. 10. At the mine itself the effect of the fault is to bring rhyolite No. 10 on the southeast side of the fault and dike against rhyolite No. 16 on the northwest side. The geological relations of the ore are diagrammatically indicated in fig. 11. The basalt dike is much broken near its walls and a considerable part of

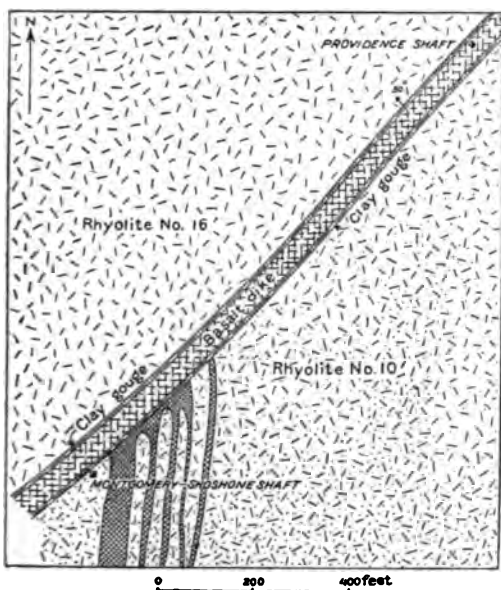


FIG. 11.—Diagrammatic plan showing mode of occurrence of ore in the Montgomery-Shoshone mine.

the faulting is later than the intrusion. How much of the total throw was effected before the injection of the dike can not be determined. In the Providence mine the basalt has been crosscut and a drift has been run along its hanging wall, which is a remarkably regular surface of rhyolite polished by the movement of faulting. All of the basalt is fractured and disturbed, and near the hanging wall the squeezed fragments have curved, glossy surfaces, recalling those common in many large bodies of serpentine.

The fissures that have determined the ore deposition in the Montgomery-Shoshone mine strike, in general, a little east of north. They appear to terminate at the basalt, since no continuation of them has been found on the northwest side of the dike. Along these fissures

the rhyolite has been altered to soft masses of kaolin separated, as shown in fig. 11, by ribs of comparatively unaltered rock. Near the basalt the kaolinized zones widen and, in some cases, coalesce. Here occur the largest and richest ore bodies, the principal one, said to average \$700 a ton, being approximately 60 feet in horizontal diameter. Along the line of the fissures southward, away from the basalt, the ore becomes of lower grade and there is less kaolinization, the soft material changing gradually into crushed and somewhat silicified rhyolite.

The determination of the relative age of the north-south fissures and of their mineralization, on the one hand, and of the basalt dike and the faulting on the other, is a problem of some practical importance. Are the fissures older than the dike and have their northern continuations been displaced by the great northeast-southwest fault? Or are they younger than the dike and have they no continuation beyond it? Unfortunately the careful study that might have afforded some answer to these questions was rendered impossible at the time of visit by the attitude of the company then in control of the mine. It is thought, however, that the mineralization and probably the fissuring along which ore deposition took place are younger than the dike. The absence of ore from the basalt itself and from the main fault, except where the north-south fissures meet it, offers no serious objection to this view, whereas the coalescence of the ore bodies and the increase in their size and richness near the dike are distinctly in its favor.

#### NATIONAL BANK.

As exposed at the surface and on the 100-foot level, the National Bank vein is a rather indefinite zone of fissuring in rhyolite No. 5. It strikes about N. 15° E. and dips west at 60°. The hanging wall is a basalt dike with an average width of 3 to 4 feet. The lode has no recognizable foot wall and consists of somewhat silicified rhyolite cut by many small and inconspicuous veinlets of quartz. The gold is not confined to the veinlets, but occurs also in the rhyolite, particularly where the latter shows little limonitic specks representing oxidized pyrite crystals.

The ore is bunched and good assays are sometimes obtained from the rhyolite at a distance of 30 or 40 feet from what is considered the main lode. The best assays obtained prior to the date of visit were about \$230 a ton, mostly in gold. In the lower grade ore the silver may exceed the gold, in one case there being about 5 ounces of silver to 3 of gold.

Considerable work has been done on the 200-foot level, mainly on a narrow seam which dips eastward at an angle of 45° to 50°, and which is probably the contact between two rhyolitic flows. The dip of the National Bank lode from the surface to the 100-foot level would, if

continued to the 200-foot level, carry the vein from 50 to 60 feet west of the shaft, or from 15 to 20 feet west of any crosscut in existence on this level at the time of visit.

#### GIBRALTAR.

There are six veins recognized in the Gibraltar property. Their strikes range from N. 15° W. to N. 20° E. and they dip to the west. The veins are numbered from east to west. No. 4, on which most work has been done, strikes about N. 10° W. and dips west at angles varying from 45° to 55°. No. 2 vein is the most nearly vertical, its dip at the surface being about 80°. The croppings of the veins are spaced at various distances apart, ranging from 50 to 150 feet.

The general country rock of the mine is a rhyolitic flow breccia containing occasional inclusions of basalt up to 3 feet in diameter. It has been correlated with rhyolite No. 6, although the many faults in Bonanza Mountain render the interpretation of the structure less satisfactory than in other parts of the district. The rock is considerably altered and contains much secondary quartz.

The veins are small and occupy fault fissures of slight throw. No. 4 vein, which has been opened by two tunnels and has supplied all the ore so far found, is in most places less than a foot wide, although local widths of 3 to 4 feet occur. It consists of quartz and a soft, dark, earthy material, which is in part oxide of manganese. A cavernous and platy structure, due to the solution of carbonates from an originally banded vein, is common. The best ore lies near the foot wall and usually includes a little of the rhyolite. The gold, which contains so much silver as to constitute electrum, occurs in the mode characteristic of the district, usually in little limonitic specks in quartz or silicified rhyolite. These rusty specks in rare instances show small residual grains of pyrite.

The veins in general cut two older structures in the rhyolite. One of these is a set of planes dipping east at angles ranging from 30° to 35° and parallel to the flow bands in the rhyolite. These may be partings between separate flows, or possibly cracks formed by the cooling of the rhyolite. The other structure is a fairly regular sheeting striking N. 30° W. and dipping southwest at an angle of 60°. When the lode fissures were formed they appear to have occasionally followed one or the other of these earlier fissures for short distances, so that the lodes are unusually crooked, as may be well seen in No. 3 tunnel on No. 4 vein.

#### ECLIPSE AND TRAMP.

The principal veins in these properties are the Hobo, Lester, and Eclipse. Their general relations may best be understood from fig. 12, for which no pretense of accuracy is made. The Hobo shaft, situated



on the crest of Bonanza Mountain, is an incline on the Hobo vein, which at this point appears to strike a little west of north and dips west at angles ranging from  $50^{\circ}$  to  $55^{\circ}$ . Just north of the shaft the vein is cut by a fault, marked by a smooth, regular slip plane which strikes N.  $10^{\circ}$  E. and dips west at nearly the same angle as the vein. Too little work has been done to determine how this fault affects the Hobo vein, but the latter shows much disturbance in its vicinity, and is probably cut off obliquely north of the shaft. The same fault can be traced northward along the east slope of Sutherland Mountain and over the slopes east of Busch Peak. Little masses of basalt occur here and there along its course, and it probably represents an old fissure which, after being filled by a dike, became again a zone of faulting. Thus the Hobo vein may never have extended north of the point where it now terminates against a plane that, while it shows at least some movement since the vein was formed, may also have been a barrier

to solutions when the ore was being deposited. The conditions, in other words, are similar to those at the Montgomery-Shoshone mine.

South of the Hobo shaft the vein can be followed for a short distance along the ridge and then it turns down the west slope of the mountain toward the mouth of the tunnel shown in fig. 12. On the ground this curve is exaggerated by the relation of the dip of the vein to the slope of the hill. Near the portal of the tunnel the fault and the vein again come together. South of the tunnel a very large vein continues along the west slope of

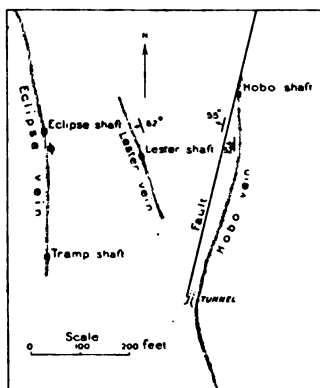


FIG. 12.—Sketch plan of the veins of the Eclipse and Tramp groups.

Bonanza Mountain and down the south slope toward Bullfrog. This vein, as may be seen from fig. 12, has the general course of the Lester vein, but it appears to be continuous with the Hobo vein.

As exposed in the Hobo shaft the vein consists of a broken mass of quartz, calcite, and earthy oxide of manganese, the last being a residue from the solution of a manganiferous carbonate and the quartz and calcite being partly secondary. The vein is small near the surface, but becomes larger and more disturbed with depth. The best ore, which affords assays up to \$400 a ton, is a fine-grained, opaque, white quartz in which free gold occurs in small limonitic specks. South of the tunnel the vein is less disturbed, and in places shows from 25 to 50 feet of fissured and shattered rhyolite full of banded veinlets of quartz and calcite. This part of the lode is apparently of low grade.

The Lester vein is a zone of fissured and veined rhyolite a foot or two wide, with a regular hanging wall. It strikes N.  $18^{\circ}$  W. and dips

west at 52°. An inclined shaft 60 feet deep has been sunk on the vein, but no shipping ore has yet been found.

The Eclipse vein coincides with an important fault which passes through the saddle north of Sutherland Mountain. The general course of the fissure is thus nearly north-northwest, but between the Eclipse and Tramp shafts the vein runs north and south. At the Eclipse shaft the lode, which is practically vertical, has a maximum width of 12 feet. Samples across this width are said to give assays of \$34 a ton. The vein is similar in general character to the Hobo and has no very definite walls. The lode itself, however, is traversed by a smooth and regular plane of faulting.

#### DENVER.

The Denver vein occupies part of a fault or fault zone that can be traced northward over Busch Peak. The fissure that has hitherto furnished the ore strikes from N. 5° to 10° E. and dips east at angles ranging from 65° to 70°. This, however, is merely one of several branching fissures, some of which strike nearly northwest. All the fissures traverse rhyolite No. 5, which is much cracked and somewhat silicified in their vicinity. The Denver lode is generally similar in character to the Hobo and the Eclipse. The streak of shipping ore varies in width up to a maximum of 18 inches.

#### ORIGINAL BULLFROG.

The Original Bullfrog lode outcrops more conspicuously than the other deposits in the district. It is a huge mass of nearly solid quartz, which, as a whole, dips north at 18° to 20° and is at least 60 feet thick. This quartz rests in most places upon a much sheared shaly material, greenish or reddish in color, which was probably originally a glassy rhyolitic flow. It is now so soft and altered, however, that its original character must remain somewhat in doubt. This material is not of great thickness and in some places the quartz rests directly upon the Paleozoic limestone. The lode has no definite hanging wall, but is overlain by rhyolite No. 2, which is fissured and contains stringers of quartz for some distance above the mass of the lode. The deposit represents a mass of rhyolite that has been greatly fissured and shattered. The fissures have been filled with quartz and with minor amounts of calcite and ore minerals, and to a considerable extent the shattered rhyolite has been completely silicified.

Some bunches of rich ore have been found, but the mass as a whole is of very low grade. The fissuring that provided opportunity for the deposition of so much silica was probably caused by movement along the fault which has here brought the rhyolites against the pre-Tertiary rocks. A deposit having some analogous features with the Original Bullfrog and known as the Wildcat occurs about a mile southeast of

Beatty on the line of the fault between the rhyolitic series and the older rocks. Here, however, it is the limestone that is irregularly silicified and stained with malachite. The ore, which is apparently only a small bunch, carries silver and a little copper. It is noteworthy that the only occurrences of copper minerals noted in the district should be situated so far apart on the same zone of faulting between the rhyolites and the pre-Tertiary rocks.

#### GOLD BAR.

The Gold Bar lode is a zone of irregularly fissured and brecciated rhyolite fully 100 feet wide. The hanging wall is fairly regular and persistent and is apparently due to a slip later than the formation of the lode. No foot wall has yet been found. The general strike of the hanging wall is N. 50° E. and the dip is northwest at 65°. Most of the stringers making up the lode are filled with quartz, although calcite and siliceous pseudomorphs after calcite occur, particularly near the hanging wall.

The best ore occurs near the hanging wall and is said to average about \$30 a ton, but occasional small bunches are very much higher in grade.

#### HAPPY HOOLIGAN.

At the Happy Hooligan mine, limestone, probably Paleozoic, is overlain by a flow of basalt estimated to be 40 to 50 feet in thickness. The basalt is in turn overlain by a much thicker flow of rhyolite. The surface of the limestone is hummocky, slopes of 20° to 30° being not uncommon. A little oxidized sedimentary material occurs at some points between the basalt and the limestone.

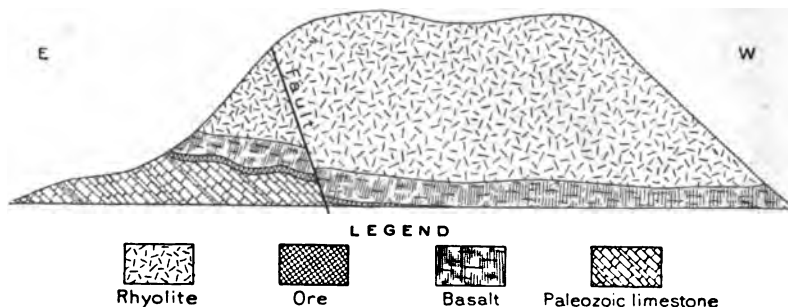


FIG. 13.—Diagrammatic section through the Happy Hooligan mine.

The ore forms a thin, soft undulating blanket deposit between the basalt and the limestone and consists of a waxy white and brown material (probably kaolin, although it has not yet been chemically examined) carrying free gold. Some of the ore appears to be altered basalt and some may represent the alteration of tuffaceous material that lay on the limestone when the basalt was poured out. The ore is rarely more than a few inches in thickness.

The deposit outcrops along the east slope of a small hill and has been followed westward by a tunnel to a point where the ore is cut off by a fault. The fault plane dips west at an angle of 60°. A winze on the fault was 20 feet deep at the time of visit and showed a basalt hanging and limestone foot wall. The structural features of the deposit are diagrammatically shown in fig. 13.

The source of the gold that has been locally concentrated along the contact between the Tertiary volcanic rocks and Paleozoic limestone is not known. The gold is flaky and has none of the characteristics of placer gold. The character of the matrix, moreover, suggests deposition from solutions capable of effecting considerable alteration in the rocks traversed by them.

No ore has yet been shipped, and future developments depend largely upon the conditions that may be disclosed by the winze now being put down on the fault.

## SEARCHLIGHT AND ELDORADO DISTRICTS.

### INTRODUCTION.

The Searchlight and Eldorado districts were visited early in February, 1906, partly in response to a petition from all of the principal mine owners or mine managers to the Secretary of the Interior asking for detailed geological work in the vicinity. The main object of the visit was to procure such general information as would enable future topographic and geological work to be laid out to the best advantage. In all about four days were spent looking over the Searchlight district and one in Eldorado Canyon. The following notes are necessarily fragmentary, but may be worth recording as a preliminary sketch of a region concerning which very little has been published and in which active development is now in progress.

### SITUATION.

The Searchlight district is in the extreme southern point of Nevada in Lincoln County, 12 miles west of the Colorado River and about 50 miles north of Needles. Two stage lines, approximately 25 miles in length, connect the town of Searchlight with the nearest railway stations in California, which are Nipton, on the San Pedro, Los Angeles and Salt Lake Railroad, 56 miles south of Las Vegas, Nev., and Manvel, on the Ivanpah branch of the Atchison, Topeka and Santa Fe system, 30 miles north of Blake on the main line. Railways are projected from both Nipton and Manvel into Searchlight.

Eldorado Canyon heads about 22 miles north of Searchlight and is 10 miles in length. It extends east and west, embouching on the Colorado River. There are no regular means of communication with the Eldorado Canyon district, which, however, is connected by a good road with Searchlight. A weekly mail service

was formerly maintained between Chloride, the terminus of the Arizona and Utah Railroad, which connects with the Santa Fe system at Kingman, Ariz., but whether a stage was running over this route early in 1906 could not be ascertained.

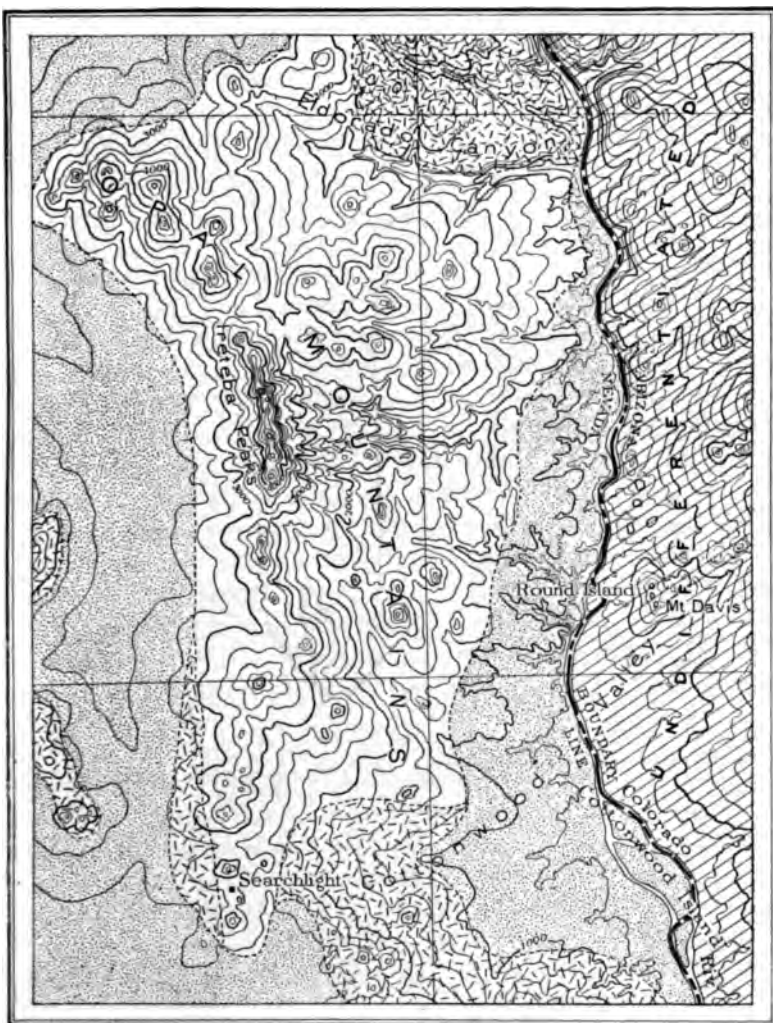
No recent surveys have been made of this part of Nevada, and existing maps are all more or less obsolete and inaccurate. On most of them the town of Searchlight is either not shown or is only approximately located. The general positions of Searchlight and Eldorado Canyon are indicated, as nearly as can be determined, in the accompanying index map (fig. 1, p. 9), and the relations of the two districts to each other and to the Colorado River are shown in the sketch map forming Pl. IV.

#### HISTORY.

Mining in Eldorado Canyon is said to date from about the year 1857, and the Techatticup (an Indian name, said to signify "plenty for all"), one of the principal mines, is reported to have been in intermittent operation since 1863. In 1883 the same mine was worked with 23 men, the ore being treated in a 15-stamp mill. The district bears evidence of having been extensively worked for many years past, large bodies of ore having been stoped from the Quaker City, Mocking Bird, Wall Street, and Techatticup mines. Latterly, however, the only work on this group, now under one ownership, has been confined to prospecting the Techatticup lode below the old stopes.

It is a little surprising that a district once alive with activity should have attracted so little outside notice. This, however, is partly accounted for by the overshadowing predominance of the Comstock, Eureka, Ely, and other districts noted in the early history of mining in Nevada and by the isolation of Eldorado Canyon. The rich ore shipped from the canyon in early days was taken down the Colorado by boat to Needles, Yuma, or the Gulf of California.


The Searchlight district is of comparatively recent development. Ore was discovered early in 1898 on the Searchlight claim, now part of the Duplex mine, and some of the richest ore found in the district came from the stopes above the 200-foot level on this lode. The principal mine at present, the Quartette, was opened in the same year, and in 1902 a 20-stamp mill was erected on the Colorado River. This was shortly afterwards connected with the mine by 15 miles of narrow-gage railway. In June, 1903, after the mill had been running about six months, the miners in the district struck, and after three months' idleness the Quartette mine was reopened with nonunion men. About this time water was encountered in the lower levels and this led to the construction of a new 20-stamp mill at the mine and the abandonment of the project for milling at the river.



Scale  
0 1 2 4 6 8 10 miles

Contour interval 250 feet

  
Alluvium

  
Quartz monzonite

  
Tertiary volcanic  
rocks

GEOLOGIC SKETCH MAP OF THE SEARCHLIGHT AND ELDORADO DISTRICTS.



## PRODUCTION.

The total output of the Eldorado district is not known and is difficult to estimate. It may be anywhere between \$2,000,000 and \$5,000,000. The Techatticup mine is said to have produced \$1,700,000, but this estimate could not be verified. The production of the Searchlight district to the end of the year 1905 probably lies between \$1,750,000 and \$2,000,000. Of the total sum the Quartette mine has produced approximately \$1,250,000.

## TOPOGRAPHY.

The Eldorado and Searchlight districts both lie in a range of hills known as the Opal Mountains and locally marking the edge of the diversified plateau that in this region borders the valley of the Colorado on the west. The general altitude of this plateau as defined by its broad desert plains is from 2,000 to 3,000 feet above sea, or from 1,000 to 2,000 feet above the river. The town of Searchlight is approximately 2,200 feet above the Colorado, which here flows in a valley whose width varies from 10 to 15 miles, although very little of this is occupied by actual flood plain. At Searchlight the hills rise to a maximum height of about 1,000 feet above the alluvial plains and are not particularly rugged. At Eldorado Canyon the general slope from the plateau edge to the Colorado River is rather steep and is deeply scored by erosion, so that contrasts in relief are more marked than at Searchlight.

## GEOLOGY.

In the very brief time spent in these districts only the most obvious and general relations of the rocks could be ascertained. The accompanying geological sketch map is merely a rough diagrammatic representation of the apparent distribution of the rocks, and the contacts as sketched may be a mile or more from their actual positions. Such a sketch, valueless as to details, will, however, if its limitations are clearly understood, assist the reader in understanding the broad features of the geological structure.

The essential facts of this structure are the existence of a north-south belt of quartz monzonite (locally known as granite), gneiss, and schist, which forms the main mass of the Opal Mountains and is flanked here and there by areas of Tertiary volcanic rocks. The schists and gneisses are the oldest rocks in the region and may be pre-Cambrian. They are much disturbed and are cut by the quartz monzonite as well as by dikes of aplite, pegmatite, and andesitic porphyries. Near Searchlight these older rocks are represented by biotitic gneiss forming part of the ridge that extends south of town and east of the Quartette mine. North of the town for a distance of 15



miles the Opal Mountains appear to consist mainly of quartz monzonite, with possibly some granite. At the Nob Hill camp, however, situated on the crest of the ridge, 4 or 5 miles south of Eldorado Canyon, the prevailing rocks are micaceous and hornblendic schists and biotitic gneisses. Between Nob Hill and the head of Eldorado Canyon are rounded hills of gneiss and schist, cut by irregular dikes of pegmatite, aplite, fine-grained quartz monzonite, and various andesitic porphyries. These hills here mark the edge of the general upland, which descends in a rugged canyoned slope to the river. So far as could be seen in the foreground of the grand panorama spread out eastward from Nob Hill the schists and gneisses extend to the Colorado on the south side of Eldorado Canyon. The upper part of Eldorado Canyon, particularly the north side, is chiefly in quartz monzonite.

The "granite" in Eldorado Canyon and near Searchlight is uniformly a fine-grained gray rock, showing to the naked eye abundant biotite, some hornblende, and apparently considerable quantities of plagioclase as well as orthoclase. Titanite is a noticeable accessory mineral. The quartz is very inconspicuous, and the general appearance of the rock suggests a fine-grained monzonite rather than a true granite.

This suggestion is corroborated by the microscopic study of thin sections. These show that plagioclase and orthoclase are about equally abundant. Quartz is a more important constituent than would be supposed from a cursory examination, it filling many of the interstices between the partly idiomorphic feldspars. The principal dark constituent is biotite, which, however, is in most places accompanied by a little green hornblende and a colorless monoclinic pyroxene. Titanite, apatite, and magnetite are all fairly abundant accessory minerals. The plagioclase, although probably not all of the same composition, corresponds in general to andesine. An estimate of the volumetric proportions of the various minerals in a specimen from Eldorado Canyon afforded a basis for a calculation of the following approximate mineralogical composition of the rock by weight:

*Approximate mineralogical composition of quartz monzonite.*

Andesine .....	34.7	Augite .....	2.3
Orthoclase .....	34.0	Titanite .....	.8
Quartz .....	12.6	Apatite .....	.5
Biotite .....	6.9		
Magnetite .....	4.4	Total .....	100.0
Hornblende .....	3.8		

The rock is thus, in all probability, a quartz monzonite, intermediate in composition between a true granite and a quartz diorite.

The quartz monzonite, like the gneiss and schist, is supposed to be *a part of the old crystalline floor* through which the Tertiary eruptives

broke. Definite proof of this, however, was not obtained in the course of the present reconnaissance, and the possibility of its being a Tertiary intrusive mass is still open to consideration.

Bordering the quartz monzonite and schist belt on the east, near Searchlight, and lying between these rocks and the river, is a zone of volcanic rocks several miles in width. East of Searchlight the volcanic series is several hundred feet thick and consists of flows of hornblendic andesite, beds of andesitic breccia, and white and brown tuffaceous sandstones and some minor flows of basalt. The whole series of effusive lavas and tuffaceous beds dips to the west, the maximum dip observed being  $55^{\circ}$ . This general westerly dip is maintained close up to the quartz monzonite (fig. 14), and it is difficult to avoid the conclusion that the contact is a fault. No definite fault plane, however, could be discovered in the brief time devoted to this area, and along the general line where the fault might be expected there are usually irregular intrusive bodies of a biotitic andesite porphyry, which, on account of its conspicuous white phenocrysts of feldspar, is

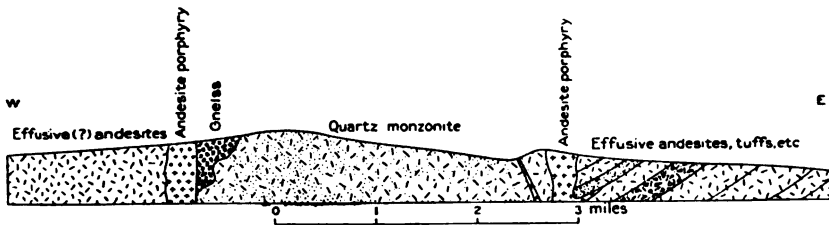


FIG. 14.—Diagrammatic east-west section across the Searchlight district.

locally known as "bird's-eye porphyry." It is possible that the volcanic rocks were poured out or deposited in a basin bounded on the west by the quartz monzonite and schist ridge of the Opal Mountains, but the high dip, the apparent steepness of the contact, and the presence of the intrusive andesite porphyry suggest that the quartz monzonite has been relatively upthrust by faulting and that intrusions of porphyry took place along the zone of dislocation.

On the west side of the quartz monzonite, near Searchlight, the relations of the rocks appear to be generally similar to those just described, although they are even less clearly shown. West of the monzonite and gneiss are low slopes of andesite passing westward beneath a cover of alluvium and traversed near the granite by numerous dikes of andesite porphyry. The same porphyry has also invaded the gneiss south of Searchlight in a multitude of nearly parallel dikes, in a way that will be more fully described in connection with the Quartette mine. Whether the hornblendic andesites west of Searchlight are flows or intrusive masses is a question that only detailed study can satisfactorily answer. They are probably flows.

On the north side of Eldorado Canyon volcanic rocks occur in great thickness between the abandoned town of Nelson, about 7 miles from the river, and the mouth of the canyon. The lowest member of the series is a tremendous flow of purplish-red, oxidized basalt, fully 1,000 feet in thickness and vesicular throughout. This rock forms picturesque cavernous cliffs just north of Nelson. It is overlain by a nearly equal thickness of tawny and buff rhyolite flow breccia. These flows seem to be entirely later than the mineralization in the upper part of the canyon and may be younger than the andesites near Searchlight. They evidently filled a valley in the older rocks, since the rhyolite between Nelson and the Techatticup mine (a mile or two east of the town site) rests directly upon an uneven surface of granite. These flows, unlike those east of Searchlight, dip toward the river.

#### MINES OF THE SEARCHLIGHT DISTRICT.

##### DISTRIBUTION.

The principal mines and prospects in the Searchlight district are in a north-south belt, about 4 miles long and three-fourths of a mile wide, which lies just west of the town. On the east side of the ridge, between Searchlight and the river, are a number of scattered prospects, none of which has yet produced important quantities of ore.

##### GENERAL CHARACTER OF THE DEPOSITS.

With the exception of the Southern Nevada, the mines of the belt west of Searchlight that were visited (and probably also those not entered) are on fissure zones striking approximately N. 65° W. and dipping southwest at various angles. All are within half a mile of the general contact between the quartz monzonite and gneiss on the one hand and the Tertiary eruptives on the other. In some mines, such as the Quartette and possibly the Duplex, the lode crosses the contact and extends into the area of the older rocks.

The lodes contain very little solid quartz and do not outcrop prominently. Toward the west they either pinch out or pass beneath the alluvium that covers the lower slopes of the hills. They are essentially mineralized fault zones in which the original character of the mineralization has been obscured by repeated movement and by oxidation. The lode material is generally a soft mass of shattered or crushed country rock colored by chrysocolla and oxides of iron and carrying free gold as its valuable constituent. Quartz is common in vugs, druses, and veinlets. Cerussite is a characteristic constituent of the Quartette ore and is associated with wulfenite, cuprodesclowitzite, and leadhillite. Other lode minerals are specular hematite, mala-

chite, azurite, and calcite. The only sulphides noted are chalcocite, galena, and pyrite, which are found in small quantities in the Quartette mine.

The material thus far stoped is almost wholly free-milling gold-silver ore. It is evident, however, that below the zone of oxidation ores may be expected that will not yield to direct amalgamation.

#### NOTES ON INDIVIDUAL MINES.

##### QUARTETTE MINE.

The Quartette Mining Company owns three groups of claims, one comprising the Golden Treasure and Copper King claims, situated about three-fourths of a mile southwest of Searchlight, another known as the Boston group, which is about  $1\frac{1}{2}$  miles northeast of town, and the third consisting of a group of comparatively undeveloped claims near Fourth of July Mountain, about 3 miles east of Searchlight. Operations are at present confined to the Golden Treasure and Copper King claims, in what is generally known as the Quartette mine.

The Quartette lode occupies a strong fissure zone striking from N.  $65^{\circ}$  W. to N.  $68^{\circ}$  W. The dip is to the south and is rather variable, ranging from  $20^{\circ}$  to  $70^{\circ}$ . The average is probably about  $50^{\circ}$ . The mine is opened by an inclined shaft on the lode, the shaft pitching somewhat to the west. The total depth is 850 feet on the incline or 600 feet vertically. There are 9 levels, rather unevenly spaced. The deposit on the whole is regular and easily followed, so that there has been little exploration of the country rock in the hanging and foot walls. Almost all of the development thus far has been to the east of the shaft, in which direction the outcrop of the lode can readily be followed over the ridge toward the Rambler mine. The vein has been opened at several points, particularly on the Copper King claim near the crest of the ridge, and in most places is easily recognized by the presence of quartz, chrysocolla, and hematite. West of the shaft the vein can be followed only a short distance.

The general geological relations of the deposit are shown in Pl. V. Prof. T. A. Jaggar, of the Massachusetts Institute of Technology and Harvard University, spent a few weeks in the district in 1903 and prepared a report on the geology of the Quartette mine. The geological boundaries shown in Pl. V were taken from a map accompanying this report. The contours, however, are sketched from memory. The contact between the andesite and the older rocks, here represented by biotitic gneiss with some aplite dikes, is marked by a ravine just east of the mine buildings. East of this ravine rises the main ridge south of Searchlight, composed of gneiss which is cut by a multitude of nearly parallel dikes of andesite porphyry. These dikes

have a general strike of N. 65° W. and follow the foliation of the gneiss. Most of them appear to terminate at the contact between the gneiss and andesite, but some continue into the latter rock, which contains also some less regular intrusions of the same porphyry. The rocks in the vicinity of the lode, particularly the andesite, show considerable alteration, which, joined with rather poor exposures and the great number of the closely spaced dikes, renders the working out of the structure a matter of some difficulty. The underground workings, unfortunately, do not promise much assistance in the elucidation of the local geology.

The lode varies in width, the maximum measurement being 50 feet, and is in general a soft oxidized mass consisting of shattered and mineralized gneiss, andesite, and andesite porphyry. The hanging wall is usually defined by a seam of soft, moist clay, but the rock is much broken and is seamed with veinlets of quartz and calcite for an unknown distance back of this wall. In some places there is a similar gouge along the foot wall, but as a rule this wall is not well defined. Subsidiary slips, similar to those along the foot and hanging walls, are often found in what is regarded as the body of the vein. Owing to the general oxidation it is impossible to get a satisfactory conception of the original character of the vein. In its present form it is a complex structure modified by brecciation and by more or less migration and redeposition of its mineral constituents. The country rock near the lode is in most places much crushed and altered, and it is doubtful whether, without more numerous crosscuts, the complex relations of andesite, andesite porphyry, and gneiss can be satisfactorily worked out. While the lode dips to the south, the dikes as a rule dip steeply to the north. The result is that abrupt changes in the character of the country rock are very common in the mine. No relation has so far been discovered between kind of country rock and value of ore.

As no stope maps are kept, it is not practicable to ascertain the exact shape of the pay shoots. The first discovery of ore was at the surface, about 100 yards west of the present shaft, where a rather irregular bunch was extracted which apparently had no connection with ore afterwards found in the mine. Ore occurs at the surface also at the Copper King tunnel near the crest of the main ridge east of the shaft. The main workings of the mine, however, are not yet under this point. In the mine proper there was practically no ore on the first level and very little on the second. Between the second and third levels, however, a shoot of rich ore was found east of the shaft, which pinched out 2 or 3 feet above the third level. At the shaft on the third level a body of good ore was stoped to a width of 30 feet. The shoot was short, however, and the third level as a whole has developed little ore. Below the third level the stopes are fairly continuous and wide down to the seventh level. The ore body has been





particularly productive between the fifth and sixth levels, the latter being regarded as the best level in the mine. In general the main ore shoot is about 300 feet long and up to 50 feet in width. It apparently pitches to the east. At the time of visit no stoping had been done on the eighth and ninth levels. The former had been driven about 300 feet to the east, but apparently had not reached the ore shoot, which on the sixth level is at the shaft. A good shoot of ore, however, supposed to pitch to the west, was being opened on the west drift of the eighth level. The ninth level had only short drifts and was not yet in ore. Where the ore body pinches, the lode may contract to a thin seam of gouge between walls of somewhat shattered country rock, or it may continue as a broad zone of crushed and altered rock having the general appearance of ore, but without value.

The ore is ordinarily a soft, shattered mass of altered country rock intimately mingled with the various ore minerals. It is generally oxidized, but a little chalcocite is said to be occasionally found and some residual kernels of galena were seen in the west drift on the eighth level. Good ore is nearly always associated with chrysocolla or with earthy cuprite, and in some places carries from 10 to 12 per cent of copper. Small cupriferous bunches are sorted and shipped. Where galena or, more commonly, cerussite appears the ore carries a larger proportion of silver than where copper is the dominant base metal. Wulfenite, the molybdate of lead, in characteristic square, tabular, orange-colored crystals, is very common throughout the mine and does not necessarily indicate ore. It is apparently one of the later products of oxidation and occurs in little vugs and open fissures implanted on the other minerals. Hematite is abundant, but, like the wulfenite, does not seem to be particularly characteristic of good ore. Quartz is widely distributed through the lode but never forms large or solid masses. Its most typical occurrence is in small vugs and veinlets. A little malachite and azurite frequently accompany the chrysocolla, and cerussite (carbonate of lead) is fairly abundant. Cuprodescloizite (a basic vanadate of lead, zinc, and copper) and leadhillite (a sulphato-carbonate of lead) were determined by Prof. Charles Palache, of Harvard University, in specimens collected by Professor Jaggard. Vanadinite (vanadate of lead) and native copper are reported as occasional and inconspicuous constituents of the ore. Visible specks of gold are not uncommon in the rich ore, but as a rule this metal occurs in particles too small to be seen with the naked eye.

The value of the ore varies widely. That worth from \$25 to \$40 a ton is commonly referred to as good ore and some being stoped at the time of visit had a gross value of \$400 a ton. About 75 tons of ore is hoisted daily, practically all of which is milled in the 20-stamp mill at the mine. The cost of mining and milling is between \$5 and \$6 per ton, which certainly is not excessive when it is considered that the



mine is about 25 miles from the railways and that the soft, heavy ground requires much timber, which is used in the form of squared "Oregon pine" (fir).

The Boston group of the Quartette Mining Company was extensively prospected some years ago, but the attempt to find workable ore bodies was unsuccessful. There are two shafts on the property, an incline about 300 feet deep and a well-constructed vertical shaft 400 feet deep. Both are now dismantled. The workings are close to the eastern border of the quartz monzonite, in much shattered and greatly altered andesitic porphyry, which breaks readily into small, dark-green or dark-red fragments and contains seams of epidote. The vein does not outcrop distinctly, but apparently strikes nearly east and west. Some small bunches of good ore were found, consisting of chalcocite, malachite, azurite, chrysocolla, and cerussite, but an average value of about \$6 per ton was the best that could be obtained for any considerable part of the deposit.

#### DUPLEX MINE.

The Duplex mine is situated about half a mile north of the Quartette mine, almost on the southwest edge of town. It is on a strong fissure which, like that of the Quartette, strikes N. 65° W. and dips to the south. The material of the lode resembles in general character that of the Quartette vein. The mine was not producing at the time of visit, but had recently changed hands and is likely to be extensively developed in the near future. It was the first property in the district to ship ore, and the lode has been extensively stoped from the surface to a depth of 200 feet. Some of the shipments are said to have carried as much as \$1,500 per ton. No attempt was made to examine the underground workings of this mine nor of the Good Hope mine, which is under the same management and lies between the Duplex and the Quartette.

#### SOUTHERN NEVADA MINE.

The Southern Nevada property includes two mines, one known as the Spokane, about 1½ miles north of Searchlight, and another known as the Blossom, about half a mile north of the Spokane. The 10-stamp mill and principal buildings of the company are all situated at the Spokane shaft, where the first development took place. Some good ore is said to have been found in this shaft, but when, shortly after this discovery, abundant water was encountered the shaft was utilized as a well and prospecting was begun on the Blossom claim. Here a large body of ore was uncovered, which was milled with water from the Spokane shaft, and produced about \$325,000. The company is at present endeavoring to find a profitable continuation of this ore body.

The Blossom ore body forms a nearly horizontal blanket deposit that outcrops around the sides of a low rounded hill from 20 to 30 feet below its summit. This ore has been mostly stoped out so that the top of the hill is now supported merely by residual pillars. The rock composing the hill is a more or less altered porphyry, which seems in some places to grade into the quartz monzonite and is probably merely a textural modification of the latter rock, which appears to be somewhat variable in the vicinity of the mine. Certain facies of this porphyry resemble so closely the "bird's-eye" porphyry of the Quartette mine as to suggest that something more than a hasty reconnaissance will be necessary to disclose the relations of the quartz monzonite, the intrusive porphyries, and the andesitic flows.

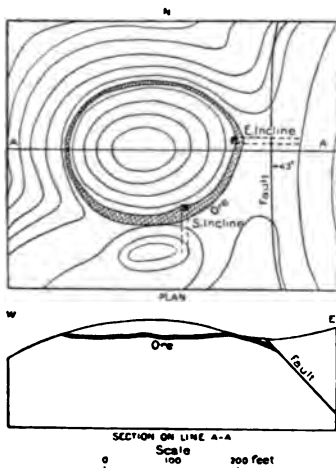


FIG. 15.—Sketch plan and section of the Southern Nevada blanket deposit.

The structural features of the Blossom ore body may be best understood by reference to the sketch plan and section of fig. 15. The main ore shoot measures about 300 feet from north to south and 250 feet from east to west. On the west and north it outcrops on the sides of the hill and beyond this outcrop has no known continuation in these directions. On the south and east, however, the ore comes to the surface and then dips under the slopes adjacent to the Blossom knoll. On the east an incline 430 feet in length has been sunk. For about 130 feet this follows what is clearly a continuation of the Blossom ore shoot, here showing a dip of  $24^{\circ}$  to the east. Below this point the incline

steepens abruptly to  $43^{\circ}$  and thence follows a fissure of entirely different character. The steeper fissure carries no ore and shows much crushing and slickensiding, accompanied by a strong and persistent clay gouge. It is filled to a width of several feet with crushed and altered quartz monzonite, shattered quartz, and fragments of dragged-in vein material. It is apparently an unmineralized fault that has cut off the ore body in this direction. In spite of its unpromising character, drifts from 200 to 300 feet in length have been run on the 130, 230, and 330 foot levels.

The south incline (fig. 15) is 220 feet in length and follows the same zone of fissuring and mineralization as that in Blossom Hill. The dip at the incline is  $35^{\circ}$  to the south, but the drifts show that the lode curves in conformity to the contour of the hill, so that a southeast dip prevails east of the incline. The intersection of the lode and the north-south fault followed in the east incline has not yet been exposed.

Some \$15 to \$25 ore has been opened up in the drifts from the south incline, but this steep part of the lode is evidently of lower grade than that stoped from the horizontal part in Blossom Hill.

The Blossom blanket varies in thickness up to a maximum of about 3 feet. It is a zone of fissured and, in some places, shattered porphyry in which the rock fragments have been silicified, and the fissures and interstices, probably once partly filled with crushed porphyry, are now occupied by masses of porous, sugary quartz in which the individual grains are small, loosely cohering crystals. The quartz is in some places solid, but such material is usually barren. There are no definite walls to the deposit, and nearly horizontal branches are common. One of these has been followed in an incline to a point 50 to 60 feet below the main stop and at this depth shows a little good ore.

As a rule the ore is not visibly mineralized. Some of the best, however, shows free gold associated with limonite and little streaks of specular hematite. No sulphides were seen, the deposit being entirely oxidized. Some of the ore left unstoped is said to be worth as much as \$55 a ton. The ratio of gold to silver varies, 1 ounce of gold to 4 of silver being apparently a common proportion.

The Blossom deposit is, so far as known, unique in the Searchlight district, although it is generally similar to some of the blanketlike lodes of Eldorado Canyon.

#### POMPEII MINE.

The Pompeii mine lies about  $2\frac{1}{2}$  miles nearly north of Searchlight and about half a mile north of the Blossom. The main lode strikes N.  $65^{\circ}$  W. and is supposed to dip about  $45^{\circ}$  SW. An inclined shaft 335 feet deep has been sunk in the foot wall of the lode at an angle of  $58^{\circ}$  and is being carried down to a depth of 600 feet. There is at present only one level, 265 feet below the collar.

The lode as exposed on this level is a brecciated zone in andesitic (?) porphyry, with a well-defined clay gouge on the foot wall and no definite hanging wall. The porphyry is much decomposed and oxidized, crumbling in many places like sand. Within the lode it is traversed by little veinlets of calcite and rusty quartz, many of them only a fraction of an inch in thickness. These little seams, particularly if soft and darkened by oxide of manganese, are said to carry such values as have been found in the lode. A crosscut, perhaps 25 feet in length, into the hanging wall shows a second lode containing considerable calcite, within which are embedded fragments of porphyry. Not enough work has yet been done to show the direction and extent of this lode, which apparently has no definite walls.

The Pompeii mine has produced no ore, although samples across 22 feet of the lode are said to have averaged \$23 per ton.

Water is abundant below the 265-foot level and is increasing as the shaft goes down. At the time of visit a Cornish pump driven

by a gasoline engine was raising 65,000 gallons in each twenty-four hours. Although this shaft was then 75 feet below the water level, the lode still maintained its oxidized character.

#### OTHER MINES.

Among the mines which could not be visited in the short time available are the Cyrus Noble, about half a mile west of Searchlight; the Searchlight-Parallel, about the same distance north-northwest of the Cyrus Noble; the Santa Fe, about two-thirds of a mile northwest of Searchlight, and the Searchlight mine, just south of the Blossom. The Cyrus Noble and Searchlight have recently erected 10-stamp mills and were preparing at the time of visit to hoist ore. The other properties are still in the prospect stage.

#### UNDERGROUND WATER.

When operations at Searchlight first began the only water near at hand was a small flow at Summit Spring, 2 or 3 miles east of the town. Later water was reached at 50 feet in Hall's well, near the spring, and at 60 feet in the Red Iron shaft, which was then utilized as a well. In the Spokane shaft of the Southern Nevada Company water was encountered at a depth of about 200 feet. The bottom of this shaft is only 4 feet below the original water level, but the supply has not noticeably diminished after about four years' pumping for milling and domestic purposes. In the Quartette mine water was reached 180 feet vertically below the collar of the shaft, and when the sixth level was opened over 12,000 gallons were pumped daily. The flow has increased with depth to the present time. The Good Hope shaft entered water at about 200 feet, and the Duplex at about 270 feet in vertical depth, the collar of the former shaft being 30 feet and of the latter 50 feet above that of the Quartette. The Pompeii shaft reached water 225 feet below the surface and at the time of visit was pumping 65,000 gallons daily. The water supply of the town is derived from a well 300 feet deep sunk in the quartz monzonite within the town. The bottom of the well is 60 feet below the water level.

The records show that in the vicinity of Searchlight, exclusive of the hills that rise notably above the town, abundant water may be expected at depths ranging from 200 to 300 feet below the surface, or in the neighborhood of 2,000 feet above the Colorado River. At points such as the Red Iron shaft, which is considerably below Searchlight in a canyon leading to the river, the water, of course, is reached nearer the surface. Deep mining will necessitate heavy pumping, but if the deep ores are not free milling they will nevertheless probably need concentration, and the water can thus be put to good use.

**MINES OF ELDORADO CANYON.****DISTRIBUTION.**

With the Eldorado mines are here included those of Nob Hill, situated on the main ridge 4 or 5 miles south of the canyon proper. Here active prospecting is in progress on the Silver Legion, Combination, and other claims, most of which were worked many years ago and produced good ore near the surface. None of the workings at the time of visit were over 100 feet in depth.

In Eldorado Canyon all the mines are on the north side. The principal group is situated near the head of the canyon and probably about 8 miles from the river. Here are the Quaker City, Mocking Bird, and Honest Miner. The first two properties were extensively worked many years ago, and the Honest Miner is said to have been the claim on which gold was first found in the district. The Quaker City and Mocking Bird are now idle, but the Black Hawk Mining Company is prospecting the Rand, Honest Miner, and other claims in the vicinity. All these mines are in fine-grained quartz monzonite.

About a mile lower down the canyon is the Wall Street mine, now idle, but with huge open stopes testifying to former activity. About 2 miles east of the Wall Street, or a mile east of Nelson, is the Buster mine, visible from the road down the canyon, and just over a ridge from the Buster, about half a mile northeast of it, is the Techatticup mine.

**GENERAL CHARACTER OF THE DEPOSITS.**

The ores at Nob Hill occur in east-west fissures in gneiss and schist. The dip is in most cases to the south and ranges from 70° to 90°. The veins are rather narrow and show no gouge and no evidence of movement since the deposition of the ore. In the Silver Legion mine the maximum width of the old stopes is about 5 feet. Although small, some of the veins are very regular and persistent, the Silver Legion, for example, being easily traceable over the surface for at least 5,000 feet. The sulphide ores, which in these veins are reached at 75 feet or less from the surface, consist of pyrite, galena, and sphalerite, in a gangue that is partly vein quartz and partly metasomatically altered gneiss or schist. The gold and silver present are probably inclosed in the sulphides, no native gold being visible. Most of the ore extracted in early days was oxidized, and contained free gold and horn silver. The Combination mine, 100 feet deep, is said to have produced about \$150,000 from ore that was floated down the Colorado on flatboats.

The deposits near the head of Eldorado Canyon are notable for their diversity in dip. All are essentially mineralized fissure zones in

quartz monzonite, but while some are almost horizontal and would ordinarily be called blankets, others are nearly vertical lodes. At the Quaker City there are two parallel, nearly horizontal veins about 100 feet apart, the upper one being exposed along the hillside for at least 1,200 feet. The average dip of these veins is  $15^{\circ}$ , in a direction a little east of north. They have been extensively stoped, in many places to a height of 7 feet, so that one can walk with ease over the smooth, slightly inclined floor of the great subterranean chamber thus formed. The ore consists of shattered quartz monzonite which has been cemented and partly replaced by calcite and quartz. Originally the deposit contained finely disseminated pyrite, but this is now oxidized and the material is a free-milling gold ore. In the Mocking Bird mine, about a quarter of a mile northeast of the Quaker City, the vein strikes N.  $25^{\circ}$  W. and dips northwest at an angle of  $40^{\circ}$ . It is in places 6 to 7 feet wide and resembles generally the Quaker City lodes. Quartz, however, is more abundant, and some parts of the vein are made up of solid white quartz inclosing fragments of quartz monzonite. This mine also contains large stopes, although development was apparently never pushed to any considerable depth.

Little could be ascertained concerning the value of the ore formerly extracted from these mines, but it is very improbable that ore worth less than \$20 a ton could have been handled under the adverse conditions that must have prevailed when the mines in the canyon were most active.

On the Rand claim, near the Quaker City mine, a prospecting shaft is now being sunk and at the time of visit was 50 feet deep. The vein strikes N.  $70^{\circ}$  E. and dips north at an angle of  $80^{\circ}$ . The vein is tight, and consists of little bunches and veinlets of quartz, calcite, and pyrite in a somewhat pyritized quartz monzonite. The lode has produced some oxidized ore from old workings near the surface, but the value and dimensions of the sulphide ore bodies are yet to be determined. The Honest Miner claim, lying a short distance east of the Rand and Quaker City, is also being prospected. Here there are two nearly parallel northeast-southwest veins about 60 feet apart and dipping about  $45^{\circ}$  to the northwest. Some ore was formerly taken from the upper parts of these veins and treated in a 1-stamp mill with cyanide tanks improvised from barrels. The veins show the usual association of quartz and calcite with specks of limonite, and are in quartz monzonite.

The Wall Street mine lies a mile or two east of the Quaker City and Mocking Bird on an approximately east-west vein with a dip to the south ranging from  $15^{\circ}$  near the surface to  $30^{\circ}$  at a depth of 30 or 40 feet. A large body of ore extending from the surface down to the water level was stoped here in the early days of the camp. The distance from floor to back is in some places fully 20 feet. No timber

was used, the back being supported by pillars of ore, and the open stope, resembling as it does a great natural cavern, is strikingly picturesque. The water level is apparently less than 100 feet below the surface. The ore, like that of the Quaker City and Mocking Bird, is shattered quartz monzonite, with bunches and stringers of quartz and calcite.

The Buster lode, formerly worked in a small way through shafts, is about a mile east of Nelson. The vein strikes northeast-southwest and has a steep northwest dip. The ore left on the dump shows quartz and calcite, with a little oxide of manganese and specks of malachite.

The Techatticup is one of the most extensively developed mines in the district. The vein strikes N. 80° E. and dips north at about 85°. It outcrops along the north slope of a steep ridge and is reached by an adit 180 feet in length and about 250 feet below the croppings of the lode. From this adit a drift several hundred feet in length has been run on the vein and at one point a winze has been sunk to a depth of 300 feet. Operations are at present confined to this winze. Above the adit level are three or four old adits and extensive stopes, which are in many places open to the surface. East of the main adit the lode divides into two, the southern branch running nearly east and west and dipping north at 60°. Both veins have been stoped along the surface to widths varying from 3 to 5 feet.

The vein is a regular and well-defined zone of shattered quartz monzonite in which the fragments are held in a network of quartz and calcite stringers. There has been no movement along the fissure since the ore was deposited, so that the vein is closely adherent to its walls and is almost impervious to descending solutions. Consequently, the sulphides at the surface, consisting of pyrite, sphalerite, and chalcopyrite, are only partially oxidized. The sulphides are usually rather finely disseminated. As the surface ore is said to have been rich in silver, galena is probably present in parts of the vein, although none was seen at the time of visit. The proportion of calcite varies widely and is greatest in that part of the lode lying east of the adit.

The average value of the ore formerly stoped from the Techatticup mine is said to have been about \$40 per ton. Unoxidized ore from the bottom of the 300-foot winze is reported to have about the same value, but detailed or reliable information regarding this property could not be obtained on the ground.

In conclusion, it may be said that one of the most striking features about the Eldorado Canyon district is the fact that the Quaker City, Mocking Bird, Wall Street, and Techatticup mines, after having produced large quantities of ore under conditions less advantageous than those now prevailing, should be idle, or, in the case of the Techatti-

cup, be prospected in a somewhat desultory manner. Of course the best ore may have been extracted, but the impression carried away from a hasty examination, without measurements or assays, was that the development of these mines under a single management and by modern methods constitutes a problem that is well worthy of careful consideration.

#### CRESCENT DISTRICT.

The little settlement of Crescent, consisting of a dozen or more tents and wooden buildings, sprang up early in 1905 in a district that was first exploited about ten years ago. The new town is 7 miles southeast of Nipton, on the road to Searchlight. It lies near the head of a small lateral valley opening on the northwest upon the desert plain traversed by the San Pedro, Los Angeles and Salt Lake Railroad and inclosed on other sides by barren hills a few hundred feet in height.

The general country rock is gneiss, in most places showing squeezed eyelike crystals (augen) of feldspar, associated with fine-grained schists and quartzite and cut by masses of granitic rock and by dikes of some basic eruptive now altered to amphibolite.

The principal active prospect is the Big Tiger, half a mile east of town. The so-called lode seems to be in reality a roughly semicircular mass of shattered quartzite that has been recemented by quartz and carries more or less gold and silver. The quartzite is apparently a residual patch, the remnant of a syncline, which rests upon the gneiss and is cut off on the southwest by a fault. Similar patches are reported northwest and southeast of the Big Tiger and presumably are similarly related to the same fault. The quartzite is much shattered by repeated movements, shows in places a schistose structure, and may be of the same general age as the gneiss.

The best assays have been obtained near the fault and indicate values of \$10 to \$12 per ton. The deposit has been prospected upon the assumption that it is a wide, irregular lode, and no attempt has been made to explore the fault, whose presence seems to be demanded by the structure, although the actual fissure is not clearly exposed. A shaft 100 feet deep has been sunk in the quartzite a few feet east of the fault, and its bottom is apparently very near the base of the quartzite. The fissured and crushed quartzite is all oxidized and shows no indication of ore other than a little iron oxide and a few specks of malachite.

About 3 miles southwest of Crescent, on the other side of a gneiss ridge, is the Calavada property. Here also the deposit appears to be a much shattered and silicified bed or beds of quartzite resting on the gneiss and dipping gently south at an angle very little steeper than the general slope of the ridge upon which the deposit lies. The



quartzite mass is fully 1,000 feet wide from east to west, and the distance from the highest point on the slope to the point where the deposit passes beneath the alluvium of the desert is probably still greater. A shaft 40 feet deep has been sunk at one place without reaching the underlying gneiss.

The material is much shattered and in many places is a breccia of quartz fragments. It is generally rusty and varies in hardness. The deposit has been carefully and conservatively sampled over an area about 1,000 feet square for the purpose of determining its availability for extensive cyanide treatment, the proposition being to handle the material with steam shovels. These assays rarely fall below \$2 per ton, and some of them run as high as \$12. The average for the whole area is approximately \$3 per ton. The deposit is separated from the railway by 2 miles of gently sloping plain, and water can be pumped from wells sunk in a playa or "dry lake" less than 3 miles away.

The occurrence of the gold in the quartzite of the Crescent district is probably due to the favorable conditions for deposition afforded by the shattering of beds too brittle to conform without fracture to movements that merely folded or crumpled the underlying gneiss and schist. Part of the brecciation may be due to faulting. In the case of the Big Tiger, the gold-bearing solutions may have come up through the fault previously mentioned. The source of the gold in the Calavada property, however, is not apparent, although the great disturbance of the mass would indicate that here, too, fault fissures may have provided channels for the rise of auriferous solutions.

## GOLD MOUNTAIN DISTRICT.

### SITUATION.

The mines and prospects of the Gold Mountain district, which are being worked at present, are situated on the eastern slope of the Grapevine Range, 20 miles northeast of the head of Death Valley and 11 miles west of Montana, a stage station halfway between Goldfield and the Bullfrog district. The old town of Gold Mountain, on the Death Valley versant of the range, and Tokop, about 5 miles to the north, were not visited in the single day devoted to this region.

### GEOLOGY.

The higher points of the rather irregular hills that here represent the Grapevine Range rise steeply from 1,500 to 2,000 feet above the alluvial slopes at their eastern base, and are composed of an intrusive mass of biotite granite, which has a width of 2 or 3 miles from north to south and extends for an unknown distance westward. This granite is intrusive into a series of sediments, originally shales with lenses of

limestone, and probably of Cambrian age. The shales are metamorphosed into fine-grained, rather obscurely crystalline biotite schists with subordinate muscovite and hornblende schists, and the limestone is recrystallized. The metamorphism is probably due to the intrusion of the granite and becomes less intense with distance from the eruptive rock. Both granite and schists are cut by numerous dikes of aplite and by dikes of fine-grained hornblende rock, probably diorite porphyry. The metamorphic rocks form low hills to the north of the granite peaks and are overlain by a thick series of Tertiary volcanic rocks. At the base of this younger group are flows of rhyolite with intercalated tuff beds, the whole being over 500 feet thick. Overlying the rhyolitic flows and tuffs, which seem to have filled a basin and thin out to the west, is a bed of red tuffaceous material, containing abundant fragments of granite. This bed is conformably overlain by a heavy capping of olivine basalt. In some places the Tertiary volcanic rocks are evidently dislocated by normal faults of considerable throw.

#### ORE DEPOSITS.

The ores, which are gold ores with subordinate silver, are found in veins traversing the granite and schists. The deposits appear in most cases to have been originally solid tabular masses of quartz; but all show more or less brecciation due to movement since the fissures were filled. None of the workings has yet gone below the zone of oxidation, although pyrite, chalcopyrite, galena, and tetrahedrite are frequently found in the generally oxidized material.

The principal mine in the district, the Rattlesnake, owned by the Bonnie Clare Bullfrog Mining and Milling Company, is in schist about 2 miles northeast of the main granite ridge. The mine has been worked intermittently for over ten years, part of the ore having been treated in a 5-stamp mill at Thorps Well, about 2 miles south of Montana station. In all, from 3,500 to 5,000 tons of ore have been run through the mill. The mine is opened by two adits about 125 feet apart vertically. The lower adit is over 400 feet long and connects with a winze 200 feet deep, from the bottom of which extend drifts about 100 feet in total length. Most of the ore formerly mined came from the upper tunnel, the stopes extending in places to the surface. At the time of visit the mine was temporarily idle.

The Rattlesnake lode strikes nearly east and west and dips north at 75°. The country rock consists of thin-banded slaty schists, some of the bands evidently representing altered limestone or calcareous shale. The planes of schistosity, which in general appear to correspond with the original bedding, dip south at various angles, the average inclination being about 45°. The lode thus cuts the planes of schistosity at a considerable angle.

The vein has a maximum width of 4 feet. It is a fairly regular filled fissure and in places shows banding due to crustification in the quartz. Like all the veins seen in the district, however, it has been affected by later movements and is generally brecciated. It consists of quartz, somewhat stained with oxide of iron. A streak of fine soft specularite, resembling rouge, occurs along the foot wall in the upper tunnel. No sulphides were seen at the time of visit, although a little galena is said to be occasionally found. The calcareous bands in the schist have apparently exerted no influence upon the vein itself, which is free from calcite. This mineral, however, is abundant in stringers and vugs in the walls in or near the limestone bands. The value of the ore, as shown in an apparently truthful statement published by the company and based on 68 samples taken from various parts of the workings, ranges up to \$213 a ton, the average being approximately \$23. No account was taken of the silver in these assays, the amount of this metal present rarely exceeding 2 ounces per ton and being usually only a fraction of an ounce.

About  $1\frac{1}{2}$  miles southwest of the Rattlesnake are the properties of the Nevada-Goldfield and Nevada-Sunshine mining companies, operated under one management, and locally known as "Parson's camp." Here active prospecting is in progress and a vertical shaft is being sunk to a depth of 300 feet in order to explore at that depth veins upon which considerable work has been done by tunneling. There are two principal veins, both in granite and about 450 feet apart. They strike about N.  $40^{\circ}$  W. and dip northeast at angles varying from  $70^{\circ}$  to  $75^{\circ}$ . Most of the work has been done on the southwest vein, which in places is fully 20 feet wide, and is said to have been traced on the surface for a distance of nearly 2 miles. This vein, which outcrops along the steep north side of the main granite ridge, has been opened by three tunnels, with several hundred feet of drifts, and by a shaft and winze at the lowest point of outcrop to a depth of 180 feet. The new shaft is being put down midway between the two veins.

The southwest vein consists of shattered quartz mingled with crushed granite. Two persistent seams of gouge are usually referred to as the hanging wall and foot wall, although the original fissuring and veining of the granite extends in many places beyond these so-called walls, which are due to later movement. In a few places the vein has been offset from 12 to 15 feet by normal faults.

The best ore occurs usually near one of the walls, in quartz less shattered than the mass of the vein, which, as a whole, shows no conspicuous mineralization. The ore commonly shows stains of malachite and contains some cerussite and pyromorphite. In places a little galena and a gray copper mineral, probably tetrahedrite, are found associated with the prevailingly oxidized ore. Free gold is rarely

seen and no native silver has been noted. Selected samples of some of the ore have afforded assays up to \$4,600 a ton, but the vein as a whole, to the depth thus far explored, is certainly of low grade. No ore has yet been shipped.

About half a mile northwest of Parson's camp is the Oriental mine, no longer worked. This was probably the first mine opened in the district and was operated through some small inclined shafts. A single loose mass of ore from the surface is reported to have been shipped to Carson City many years ago and to have produced over 50 ounces of gold. The vein, which is in schist, strikes east and west and dips north. It is well defined at the surface, and is of the brecciated type characteristic of the district.

The Courbet mine, under the same ownership as the Rattlesnake, is situated about a mile west of the Nevada-Goldfield property. It has produced some ore and over 300 tons, said to average \$32.50 a ton, is piled on the dump. The mine was idle at the time of visit.

The development consists of two adits about 75 feet apart, the upper one being 350 feet and the lower one 400 feet in length. Both are on the general line of the vein, which strikes nearly north and south with an easterly dip of 30°. The vein, which is in granite, was originally a fairly regular plate of quartz up to 15 inches in width, carrying pyrite, chalcopyrite, galena, and perhaps other sulphides. Movement since these minerals were deposited has crushed the quartz and produced seams of gouge along the hanging wall. The crushed vein material is all more or less oxidized and contains abundant cerussite, with malachite, azurite, and pyromorphite. The ore is mainly a gold ore, although it must contain notable proportions of silver and lead.

The Gila Monster mine lies about 2 miles south of the Nevada-Goldfield, on the south slope of the same granite ridge. It is owned by the Butte Gold Mining and Reduction Company. The lode was discovered in February, 1905, and is being actively prospected by a tunnel on the vein, about 400 feet long at the time of visit, a shaft 100 feet deep above the tunnel, and a winze about 50 feet deep near the portal of the tunnel. The vein strikes east and west and dips north at 45°.

The vein is large, the whole fissured and mineralized zone being from 15 to 20 feet wide, and it can be followed along the steep hillside for several hundred feet. The original fissuring appears to have followed a dioritic dike in the granite and the lode probably consisted of one or more thick plates of quartz, with many subordinate stringers and no very definite walls. Later movement has shattered and crushed the original vein material. The ore is generally oxidized, but contains a little pyrite and galena. Copper minerals are not as abundant as in the Nevada-Goldfield and Courbet veins.

# NOTES ON THE MANHATTAN DISTRICT.

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By W. H. EMMONS and G. H. GARREY.

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## INTRODUCTION.

Manhattan, Nye County, Nev., is in latitude  $38^{\circ} 28' N.$ , about 29 miles north-northeast of Tonopah and about 13 miles southwest of the old silver camp, Belmont.<sup>a</sup> It is on the western slope of the low group of hills locally known as the Smoky Mountains, which connect the Toquima Range on the north with the San Antonio Mountains on the south and separate Big Smoky Valley on the west from the northern arm of Ralston Valley on the east. The nearest railroad point is Tonopah, with which it is connected by daily stages and automobiles.

In March, 1906, a hasty reconnaissance of the geology and mines of the district, incidental to other work in Nevada, was made jointly by the writers. At that time a large part of the country was covered with snow, and much of the surface geology was masked; however, in view of the prominence which Manhattan and the surrounding country has recently attained, it seems desirable to record such facts as were observed.

## TOPOGRAPHY.

The town of Manhattan has an altitude of about 7,250 feet above sea level, and is situated in a narrow valley followed by the old Belmont-Cloverdale wagon road, which has been one of the principal highways of the desert ever since the early sixties. The rounded hills on either side of this valley rise only 200 to 500 feet above its floor, but about  $1\frac{1}{2}$  miles to the northwest they grade into the rugged mountains forming the crest of the Toquima Range, which extends northward about 80 miles farther, paralleling the Toyabe Range to the west and separated from it by the Big Smoky Valley.

## FUEL AND WATER SUPPLY.

Owing to its greater elevation, Manhattan possesses advantages not enjoyed by mining camps in the desert country farther south. The hills surrounding the town are covered with vegetation and support a growth of scrub pine and cedar that is of great value for fuel and mine

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<sup>a</sup> Location furnished by Mr. Arthur Philbrick, United States deputy mineral surveyor, Manhattan, Nev.

timbers. Good water, sufficient for present purposes, is obtained from relatively shallow wells in the valleys, and it is believed that this supply can easily be augmented by running pipe lines from springs in the mountainous country to the southeast.

#### HISTORY.

Gold was discovered at Manhattan by John C. Humphrey and companions in April, 1905, near the southern base of April Fool Hill, about 100 feet from the Belmont-Cloverdale wagon road. The first ore found gave only low assays, but in July specimens of high grade ore showing an abundance of free gold were obtained. Interest increased as subsequent discoveries were made, and in August there was an influx of prospectors, who located most of the ground for several miles around. Attention, however, was soon drawn to other fields, where new strikes were reported, and consequently early in December the town had less than 100 inhabitants. A shipment of rich ore by way of Tonopah caused an excitement there, and people from there and from near-by camps, waiting for such an opportunity, were quick to rush to the new camp. Within a few weeks a town a mile in length was built in the valley and its tributaries, and in March, 1906, there were 3,000 people in Manhattan and the immediate vicinity. The town is agreeably located, well laid out, and has many wooden buildings, while some of stone are in process of construction. It is provided with post-office, numerous stores, banks, newspapers, assay offices, telegraph and telephone service, and stage and automobile lines.

#### DEVELOPMENT AND PRODUCTION.

At the time of the visit, in March, 1906, most of the claims were being vigorously prospected and several mines were shipping ore. Much of the ground had been let out to leasers for a period of six to twelve months, each lease having 100 feet along the lode and extending to the side lines of the claim. The leasers usually pay a royalty of 25 per cent of the gross receipts. Briggs & Evans, who owned lease No. 14 on Union No. 9 claim, had sunk a shaft on the vein to a depth of 163 feet. Most of the openings, however, were less than 50 feet deep. From many of these shafts drifts extended for a distance of 10 to 50 feet.

The production, so far as could be learned, was about \$50,000. On account of excessive freight rates and treatment charges ore averaging less than \$70 was left on the dumps, awaiting the completion of mills.

#### GENERAL GEOLOGY.

The rocks around Manhattan include slates, limestones, quartzite, granite, diorite porphyry, and rhyolite. The ore bodies so far discovered are in the sedimentary rocks.

## SEDIMENTARY ROCKS.

The slates, limestones, and quartzites which make up the sedimentary series at Manhattan are exposed for 2 or 3 miles along the Tonopah road and perhaps as far on the road to Belmont. Mr. S. F. Emmons<sup>a</sup> describes a series of slates, limestones, and quartzites at Belmont closely resembling these. Further, Mr. G. K. Gilbert<sup>b</sup> notes that the slates at Belmont contain Silurian fossils, and estimates their thickness at from 4,500 to 5,000 feet. The slates at Manhattan are probably of the same age.

Near Manhattan, as at Belmont, the sedimentary rocks are extensively metamorphosed and consist for the most part of dark, fine-grained mica schists or phyllites. The limestone layers are in most cases less than 30 feet thick, and probably will be found not to persist for very great distances. Usually they have been entirely recrystallized and in some places changed to an impure gray marble. Quartzite layers also occur interbedded with the belts of schist. A quartzitic breccia or conglomerate layer less than 20 feet thick, made up chiefly of large subangular quartz fragments, occurs near the Reilly fraction, dipping with the schists. At Manhattan the glossy or satin-like phyllites or fine mica schists predominate over other members of the series. These are present in considerable variety, but the commonest facies is one which in hand specimens appears to be composed almost entirely of biotite. Microscopic study shows that this schist consists chiefly of biotite, muscovite, and quartz. The mica forms interlocking folia in perfect alignment with the schistosity, inclosing small grains of quartz having also a pronounced though less perfect linear arrangement. In certain facies crystals of chlorite and magnetite cut across laminae of the mica in a manner which shows that they were formed after the schistosity was produced. The schistosity as a rule is approximately parallel to the bedding, as determined by contacts of limestone and slates.

## GRANITE.

Granite outcrops about 3 miles southeast of Manhattan along the road to Tonopah. It is coarsely crystalline, pink or gray, and consists chiefly of quartz, feldspar, and mica. A similar body of granite occurs to the northeast of Manhattan on the road to Belmont. Mr. S. F. Emmons,<sup>c</sup> describing relations of granite and sedimentary rocks at Belmont, says: "The metamorphic rocks where in contact with the granite are frequently highly altered and may come under the general name of quartzite." Mr. Spurr<sup>d</sup> also states that the metamorphism of the sedimentary rocks to the southwest of Belmont is connected

<sup>a</sup> U. S. Geol. Explor. 40th Part., vol. 3, p. 394.

<sup>b</sup> U. S. Geol. Surv. W. 100th Mer., vol. 3, p. 180.

<sup>c</sup> Loc. cit., p. 396.

<sup>d</sup> Bull. U. S. Geol. Survey No. 208, p. 91.

with the intrusive mass of granite. It is highly probable, then, that the granite in the vicinity of Manhattan is also intrusive in the sedimentary rocks and may be regarded as one of the principal causes of their metamorphosed condition.

#### DIORITE PORPHYRY.

About  $1\frac{1}{2}$  miles northeast of Manhattan there is a large body of diorite porphyry in contact with rhyolite. The contact occurs along the bottom of a small gulch for a considerable distance, but when this place was visited 2 or 3 feet of snow lay in the bottom of this gulch, and the relations of the porphyry and rhyolite could not be clearly made out. The porphyry is undoubtedly of later age than the slates, but whether it is contemporaneous with or of later age than either the intrusive granites or the rhyolites has not yet been determined. The diorite porphyry is brown on the weathered surface and shows a large number of light-colored feldspar phenocrysts. On a freshly fractured face the groundmass is dark green and has phenocrysts of about the same shade. Microscopic study of an altered specimen shows that the porphyry consists essentially of plagioclase feldspar (andesine and labradorite) with a large amount of calcite, serpentine, and chlorite. The serpentine and chlorite are probably the alteration products of hornblende or pyroxene, and the rock is an altered diorite porphyry.

#### RHYOLITE.

Rhyolite occurs as surface flows covering both the sedimentary rocks and the granite and also as dikes cutting the same formations. To the north of Manhattan these flows cover a vast area extending approximately to the summit of the Toquima Range, giving a vertical thickness of many hundred feet. North of Belmont this range, according to Mr. Spurr,<sup>a</sup> appears to be entirely covered by these great flows of rhyolite, which are also found in the San Antonio Mountains to the southward. The rhyolite is commonly yellow, pink, or light gray and contains many phenocrysts of smoky quartz and white feldspar, with occasionally a small flake of mica. The basal facies very often contain a large number of fragments of schist, granite, and rhyolite. Under the microscope the groundmass exceeds the phenocrysts in volume and is usually glassy, sometimes spherulitic, and frequently devitrified. The feldspar crystals are orthoclase and acid plagioclase. The quartz phenocrysts usually show embayments, due to magmatic corrosion.

The rhyolites are eruptive rocks, presumably Tertiary, which are so extensive that they completely mask the earlier rocks over the larger portion of the Toquima Range.

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<sup>a</sup> Loc. cit. p. 92.



## FAULTING.

That there has been extensive faulting in the ore-bearing rocks is shown by brecciated zones and polished striated walls exposed by mining explorations. Fractures traverse the slate, limestone, and quartzite layers in all directions, and along some of them the beds have been displaced. In one or two cases fissure veins have been slightly displaced, which shows that some faulting also occurred after the mineralization.

About three-fourths of a mile north-northeast of Manhattan a fault striking N. 50° E. and dipping southeast at 55° brings the rhyolite flow breccia into contact with the truncated edges of sedimentary beds. Movement striae on the slickensided surface dip northeast at 75°. At a point farther northeast on the strike of this fault rhyolite forms both the foot and the hanging wall, which shows that this fault was formed subsequent to the extrusion of the rhyolite.

## ORE DEPOSITS.

## GENERAL CHARACTER.

So far as developments have shown the ore deposits of the Manhattan district are confined entirely to the area of metamorphosed sedimentary rocks, which is surrounded by rhyolite, granite, and alluvium.

The ore bodies are (1) narrow, tabular lode deposits, occurring in rather continuous veins crosscutting the bedding and schistosity of the metamorphosed sediments, (2) deposits interlaminated with the schistosity, and (3) deposits parallel to bedding planes of the quartzites, shales, and limestones and replacing part or all of limestone or calcareous layers.

## FISSURES.

Most of the principal ore-bearing fissures of the district, so far as could be learned from a considerable number of compass readings, strike north or a few degrees west of north. Some mineralized fractures, however, also make considerable angles with this main belt of veins. There are also numerous minor fractures, only slightly mineralized, which form a network of intersecting planes running apparently in nearly every conceivable direction.

The dips of the principal veins range from 45° to 90°; but a majority of them are inclined to the southwest at angles ranging from 60° to 70°. On account of their undulating character some of the veins may also vary considerably in strike and dip at different points along their course.

## THE ORES.

The chief gangue minerals of the ores are quartz and calcite, but barite and fluorite are sometimes present. Gold is the principal and almost the only metallic constituent of value, although silver is present at some places in amounts varying from a trace to \$2 a ton. Small quantities of ore carrying argentiferous galena have been reported as occurring near Broncho Hill and also in the neighborhood of Central City. Iron and manganese oxides are usually present, but are of no economic value. The gold occurs usually as minute particles, too small to be detected in the hand specimen, finely disseminated through the quartz, calcite, or claylike gouge filling the veins, though there is occasionally coarse gold in small transverse stringers and small sheets of leaf gold in cracks along bedding planes or between the laminae of the crushed schists. Coarse gold, however, is an exception and usually occurs only in small bunches or pockets, very irregularly and erratically distributed. On the Mayflower and Annie Laurie claims specimens of gold-bearing quartz were found which had a flaky or schistose structure, as though a calcareous schist had been replaced by silica particle by particle.

The typical rich ore of the camp is either a gold-bearing dense white quartz, stained in places to a yellowish color by iron oxides, or a mass of porous white quartz formed by intergrown tabular crystals of quartz which are pseudomorphs after calcite. In certain instances this ore is associated with a dark-brown powder which is a mixture of iron and manganese oxides. The gold in such ore is usually not visible, even with a magnifying glass, but on crushing finely and panning a good "streak" of very fine bronze-colored gold is obtained.

The shipments of this class of ore so far have yielded returns varying from \$70 to \$300 or more to the ton, but the average value has been about \$125. Numerous assays of picked specimens gave values running from several hundred dollars to many thousands of dollars to the ton.

A medium grade of ore is also found where small breccia fragments of the country rock have been cemented into a compact mass by dense white quartz similar to the quartz forming the best ores. This class of ore usually ranges between \$35 and \$70 and hardly justifies shipment at the present high rates for haulage and treatment.

The ore too low to warrant shipping is classed as milling ore. The type of low-grade ore common to the fissures which cut across the formations consists usually of a breccia of schist fragments loosely cemented by a porous matrix of quartz or of a kaolinized or clayey substance resulting from the alteration of the crushed wall rocks. At a few places the country rock immediately adjacent to the fissures, it was reported, carries values sufficient to warrant its being mined as

milling ore. The values in this case are probably not usually due to a fine dissemination of the ore-bearing minerals through the rocks, but to thin films of ore along the numerous cracks intersecting the fractured country rock.

High-grade ore may also be obtained by sorting out portions of the veins in which the limestones or calcareous shales have been entirely or partially replaced by gold-bearing quartz. Other portions of the limestone layers which have been replaced to a less extent by quartz are leaner and are classed as "mill dirt." The milling ore on the dumps is reported to run from \$10 to \$35.

In getting ore ready for shipment most of the material from the vein is run through a screen and all the "fines" are shipped directly, while the balance is sorted and the fragments of comparatively fresh wall rock, free from quartz, are picked out, for these usually carry no values.

Treatment charges (including reduction for moisture) and haulage together amount to \$35. As a rule, however, no ore which assayed less than \$70 a ton was being shipped.

#### LODE DEPOSITS.

The lode deposits consist of roughly tabular mineral masses along well-defined fissures which cut across the schistosity and the bedding planes of the metamorphosed rocks.

Typical examples of this type are the ore bodies of the Stray Dog, Union No. 9, Little Grey, and other claims in the same vicinity. In deposits of this kind the principal part of the vein consists chiefly of a breccia of schist, quartzite, and limestone embedded in a matrix of pulverized and altered country rock which in places has been partially or entirely replaced by silica and calcite. •

The richest portion of the deposit is usually a dense, hard, white quartz, frequently stained to a yellowish color by iron, or a belt of "honeycombed" porous quartz resulting from a pseudomorphic replacement of vein calcite and barite by silica. These zones of rich quartzose ore, which vary in width from 1 or 2 inches to 2 or more feet, grade into belts of lean ore that contain abundant breccia fragments of schist and other wall rocks and have a clayey material cementing the breccia fragments in place of the quartz. This lean vein material, which is in places several feet wide, then grades into crushed country rock which often carries small values to a considerable distance from the main fracture. At present the development is not sufficient to throw much light either on the shape of these ore bodies or on their distribution along the vein. Nevertheless, judging from somewhat similar deposits in other areas, it is probable that the pay shoots of irregular size and shape will be separated from one another by portions of the vein which are lean or barren.

## INTERLAMINATED DEPOSITS.

Such deposits occur immediately southeast and also north of town, and consist either of narrow veinlike deposits, resulting from mineralization along planes of movement parallel to the schistosity, or of isolated and comparatively small lenticular bodies of mineralized quartz which have their long dimensions oriented with the laminae of the schist.

The narrow veinlike deposits are variable in extent and erratic in distribution, owing to the tendency of the veins to pinch to a mere crack and then to widen out again. The exploration for the pay shoots is made difficult not only as a result of the pinching of the veins, but also because the ore streak at some places plays out along one vein and continues across a series of smaller transverse fractures to a parallel vein several feet away and then proceeds along the latter in the same direction. These deposits consist of a porous or honeycomb quartz associated with the gouge resulting from the crushing and trituration of rocks due to movement along the planes of weakness parallel to the schistosity. The ore probably partially results from a substitution of auriferous quartz for the recrystallized calcitic constituent of gouge formed from very calcareous schists or by a quartz replacement of the calcareous schists immediately adjacent to the fracture.

The small discontinuous lenticular quartz bodies parallel to the schistosity are often overlapping, although separated by considerable areas of schist, the laminae of which bend around the lenslike masses and inclose them on all sides.

At some places splendid specimens of leaf gold occur between the laminae of the schist. Considering, however, the amount of unprofitable exploratory work necessitated by the small size and irregular distribution of the interlaminated deposits, it is more than likely that this form of deposits will prove less remunerative than those in the crosscutting fissures.

## BEDDED DEPOSITS.

The bedded deposits are roughly tabular and conform with the bedding of the country rock. Their boundaries are seldom regular in shape, and they vary in thickness from a few inches to 20 feet or more. Most of them have resulted from the partial or complete replacement of beds of finely crystalline limestone by silica, while some are belts of mineralized, crushed, and pulverized country rock, brownish-gray gouge, and quartz, following the bedding and resulting from extensive movement. Some of these belts are of considerable thickness and give satisfactory values.

During the process of replacement of the limestone or calcareous shale the waters carrying the silica in solution perhaps also brought

in the precious metals, which seem to have been deposited along with the silica, since the quartz appears to carry most of the values. Apparently before the limestone was replaced it was completely recrystallized to a cleavable mass or aggregate of large white to colorless calcite crystals, the incomplete outlines of which at many places grade irregularly into the massive limestone. Subsequently silica-bearing waters replaced the calcite crystals. Specimens collected in the Manhattan Consolidated mine illustrate well the various transition stages in the replacement of individual calcite crystals by quartz. In some specimens there appears to be only a thin film of quartz enveloping a part or the whole of a crystal. Again, the silica is found to have penetrated irregularly for short distances along a few of the cleavage planes of the calcite, while in other specimens nearly all the numerous cleavage partings are filled with silica, although calcite remains between the thin siliceous films. Still other specimens show that the calcite which formerly intervened between these films has been partially or entirely replaced by silica. In many instances the siliceous pseudomorphs after the calcite are rather porous, the silica having failed to replace all of the calcite taken into solution.

The replacement of calcite by silica has proceeded most extensively along the contacts of the limestone with other formations, or along bedding planes and joints in the limestone itself.

#### DISPLACEMENTS ALONG THE FISSURES.

A large number of fractures along which movement has occurred traverse the metamorphosed sedimentary rocks.

Evidence of movement is found in the soft, claylike "gouge" which at some places fills both barren and productive fractures. The gouge in one or two instances reached a width of nearly 4 feet, while the country rock was crushed and brecciated for several feet on either side of the fracture. Polished or slickensided surfaces showing steeply inclined ( $50^{\circ}$  to  $75^{\circ}$ ) movement striæ are also of frequent occurrence.

On account of the snow and the limited time devoted to the study of the area it was impossible to determine the amount of displacement resulting from the movements, but by detailed mapping of the most prominent and easily recognizable limestone, quartzite, and shale beds both the direction of the faults and the amount of displacement of the beds can probably be ascertained.

To what extent faulting has occurred since the deposition of the ore is at present undeterminable on account of the limited exploration along even the more promising veins. However, a fault which has caused a displacement of only a few feet cuts one of the mineralized fissures on the Stray Dog claim. Again, near the south-east boundary of Union No. 9 claim there is an irregularity in the

pay streak in which the north portion of the vein appears to have been offset 6 feet to the east. The present workings do not show conclusively, however, whether this is a fault in the vein or two parallel veins 6 feet apart with a small transverse fracture connecting the ends of the pay streaks on the two different veins.

The faults are probably normal in character, which implies a relative downward movement of the hanging wall. However, there was undoubtedly also considerable horizontal movement or thrust along the fault plane, as shown by striæ and grooves. There appear to have been two or more periods during which faults were formed. The chief fracturing was probably connected with the intrusion of the granite, though there was faulting after the eruption of the rhyolite and also possibly after the intrusion of the diorite porphyry.



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[Bulletin No. 303.]

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# OIL AND GAS FIELDS OF GREENE COUNTY, PA.

BY

RALPH W. STONE

AND

FREDERICK G. CLAPP



WASHINGTON  
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# OIL AND GAS FIELDS OF GREENE COUNTY, PA.

By RALPH W. STONE and FREDERICK G. CLAPP.

## INTRODUCTION.

Greene County occupies the southwest corner of the State of Pennsylvania, extending eastward as far as Monongahela River. It is bounded on the north by Washington County, on the east by Fayette County, and on the south and west by West Virginia. It is approximately 32 miles long and 18 miles wide, having a total area of 588 square miles. The county is included almost entirely in the Rogersville, Waynesburg, and Masontown quadrangles of the United States Geological Survey.

Greene County has produced oil continuously since 1886, and since 1890 it has also yielded a large amount of natural gas. Over 1,300 wells have been drilled for gas or oil within its boundaries. So far as known these are shown on the accompanying map of the county (Pl. I, in pocket). Gas and oil wells and dry holes are represented by appropriate symbols. The map is complete to December, 1905, when the field was last visited. Other wells have been drilled since then, and it is probable that a few dry holes have been mist. While the derrick or "rig" remains standing or after the rig is blown down and only the bull wheels remain a well can hardly fail to be found, but when not even the conductor box is left the former existence of a well may escape notice.

The object of this report is to assemble the present knowledge of the occurrence of gas and oil in this county, including a large amount of unpublished data, and to present it in a convenient form for the use of those who are interested or engaged in the oil and gas business. The report describes (1) the rocks from the surface to the bottom of the deepest well; (2) the geologic structure, with a contour map of the Pittsburg coal; (3) the oil and gas sands; (4) the oil and gas fields, with suggestions for their further development. In addition many well records are given, and triangulation stations and bench marks are described.

The feature of greatest importance is the determination and representation of the geologic structure. The depth of the Pittsburg coal below the surface has been determined and a contour map

(Pl. I) constructed to show how it lies. By this map the depth of the coal below any point in the county can be told at a glance, and the shape and location of the anticlines and synclines, a knowledge of which is of great importance in the development of gas and oil pools, is clearly shown.

#### PREVIOUS ACCOUNTS OF THE AREA.

The principal published reports which discuss the oil and gas of Greene County are Report K, Second Geological Survey of Pennsylvania, 1876, by J. J. Stevenson; the Masontown-Uniontown folio (No. 82, 1902, by M. R. Campbell), Waynesburg folio (No. 121, 1905, by R. W. Stone), and Rogersville folio (No. —, in preparation, by F. G. Clapp), of the Geologic Atlas of the United States; a brief paper entitled "Oil and gas fields of eastern Greene County, Pa.," by R. W. Stone, in Bulletin No. 225, United States Geological Survey, 1904, pages 396-412; a paper entitled "The Nineveh and Gordon oil sands in western Greene County, Pa.," by F. G. Clapp, in Bulletin No. 285, United States Geological Survey, 1906, pages 362-366, and West Virginia Geological Survey, vol. 1 (a), 1904, pages 121-123, by I. C. White. In addition there are occasional brief statements and references in newspapers and technical journals.

#### ACKNOWLEDGMENTS.

The authors wish to acknowledge their indebtedness for many courtesies received from residents of the county during the prosecution of field work and to the various oil and gas companies and their field superintendents, as well as to individual drillers, for assistance and information. Special acknowledgment is made to Messrs. Reese & Heasley and P. A. Troutman, of Waynesburg, for much helpful information; to Mr. Robert Shear, of Mount Morris, for many records, suggestions, and courtesies; and to Messrs. Ira Ross, of Dunkard, and Charles Longanecker, of Sigsbee, for information concerning wells drilled and abandoned years ago. The oil and gas companies kindly furnished records of their wells, and without their assistance the most valuable part of this work could not have been accomplished. In the field work in western Greene County the authors were aided by Mr. F. W. De Wolf, to whom credit is due.

#### COMMERCIAL GEOGRAPHY.

##### RELATION TO MARKET.

Greene County is a monotonously hilly region lying a little aside from any of the great natural transcontinental routes of travel and the transportation facilities are limited. Monongahela River, a navigable waterway, forms its eastern boundary. A narrow-gage railroad, which connects with a broad-gage road at Washington, enters *the county from the north* and terminates at Waynesburg.

Greene County is 30 miles south of Pittsburg, which has been made one of the greatest manufacturing centers of the world by cheap fuel. An abundance of coal, and especially of excellent coking coal, in the immediate vicinity early determined this point as the center of iron and steel making in this country, but the later development of natural gas as a cheaper and more convenient fuel has increased very markedly the amount of manufacturing at Pittsburg. All the iron and steel mills there use natural gas in their puddling furnaces and under their boilers. Practically all manufacturing establishments, including extensive glass factories and breweries, use gas for developing power, heat, and light, and on account of its cheapness, cleanliness, and convenience of application it is also in common household use. Much of this cheaper fuel comes from Greene County. Owing to the constantly increasing demand and the gradual failure of old wells, continuous drilling is necessary to keep up the supply. Manufacturing plants do not locate in the gas fields because it is much more economical to establish factories on a transportation line and bring the gas to them.

#### PIPE LINES.

All the oil and gas produced in Greene County is transported by pipe lines. The oil wells in the eastern part of the county do not flow, but have to be pumped, and yield but a few barrels per day per well. A common practice in pumping small groups of wells is to make steam at a central boiler house, pipe it to the engines at the wells, and pump with the walking beam. At Willow Tree some of the wells are pumped with a small mechanical device known as an air head or pump head, which is operated by compressed air. The oil from the Mount Morris, Garrison, and Whiteley Creek fields is pumped to a central station on Monongahela River, 2 miles above Morgantown, W. Va., and thence to the seaboard. It is estimated that the production of that portion of the Mount Morris field which lies in Greene County amounts to about 1,000 barrels daily. The Whiteley Creek oil field produces about 80 barrels daily and the Garrison field about 30 barrels. The production of the Fonner field, which is about 50 barrels of oil a day, is carried by pipe lines to storage tanks at Meadowlands, Washington County, Pa.

The oil wells in western Greene County are mostly pumping wells, although a few of them flow. The production recorded during the first twenty-four hours is not known to have exceeded 200 barrels in any well outside the Nineveh and Fonner fields, and the general averages for good wells in the Nineveh district are 10 to 60 barrels the first day. Throughout western Greene County there have been no such enormous yields as occurred in the Washington field in the early days. Owing to this general lightness of production the South Penn Oil Company has recently abandoned drilling in that part of the county.

Three main pipe lines carry the oil from the western half of the county to tanks at Meadowlands—a 5-inch line about 40½ miles long, extending in a direct course from Littleton, W. Va., a 6-inch pipe running from Downs station, West Virginia, northward 36 miles, and a third line following a nearly direct course from Dolls Run, West Virginia. At present there are at Meadowlands about 50 tanks, having a total capacity of about 1,467,000 barrels. The tanks are not all the same size, their capacities ranging from 12,850 to 37,300 barrels, with an average capacity of about 29,350 barrels each. The entire capacity of the tanks in the southwestern Pennsylvania field amounts to not more than 1,700,000 barrels. The yield from the various oil fields can not be readily ascertained.

Most of the gas from this area is carried by pipe lines to Pittsburg, where it is used largely by manufactories for steaming and heating purposes and by the city in general for heating and lighting. Some goes to Wheeling and other points in West Virginia, and some is used locally for heating, lighting, and cooking, as at Waynesburg and on many farms which are near producing wells.

The Carnegie Natural Gas Company has a pump station 2 miles east of Waynesburg that draws from wells in the Waynesburg field and from two 10-inch lines, one of which extends to Mount Morris and the other enters West Virginia 3 miles west of Blacksville. The gas is sent to Pittsburg from this station thru two pipe lines, 12 inches and 16 inches in diameter. This company has also smaller lines in the western part of the county. The Philadelphia Company has a 16-inch line to Pittsburg which extends across the county from northeast to southwest, a 10-inch line from Blacksville to Waynesburg, two or three lines to the Waynesburg field, and a second 16-inch line from Waynesburg northeastward to Pittsburg. The Peoples Natural Gas Company has lately completed a 20-inch line which enters Greene County at Dent, 3½ miles west of Blacksville, passes 4 miles east of Waynesburg, and crosses the county line again near Zollarsville. The Manufacturers Light and Heat Company takes gas from the field south of Waynesburg through a 16-inch line, and the Fort Pitt Gas Company has a 12-inch line which crosses the county from a point 4½ miles east of Blacksville to Zollarsville and carries gas to Pittsburg. The Natural Gas Company of West Virginia and the Wheeling Gas Company have several 8-inch and 10-inch lines in the western part of the county which carry gas to Wheeling and other points on the Ohio River.

#### STRATIGRAPHY.

##### GENERAL STATEMENT.

The rocks exposed at the surface of Greene County are about 1,400 feet thick. The section includes the upper part of the Conemaugh formation, which outcrops in a narrow belt on Monongahela River,

the Monongahela formation, and the Dunkard group. It happens that the Pittsburg coal, which is at the base of the Monongahela formation, is the lowest easily recognized bed outcropping in the county and is also the stratum used by drillers throughout the county for calculating distances to productive sands. It is encountered at the surface and at varying depths down to more than 1,300 feet. Being confident of finding and recognizing it, drillers often neglect to keep detailed records of the overlying rocks. For this reason, and because this bulletin is designed primarily for the use of oil and gas men who think of the rocks in the order in which they find them, it seems advisable to divide the discussion of the stratigraphy of the county into two parts, considering first the rocks exposed, or all above the Pittsburg coal, and then the rocks below the surface, or lower than the Pittsburg coal, and thus to treat the rocks from the driller's point of view, as they are discovered by the descending drill. A description of the surface rocks is pertinent to this report because, though the Pittsburg coal outcrops along Monongahela River, throughout much of the county it is several hundred feet below the surface. If the driller can recognize the horizon which his hole has reached by the character and sequence of the higher beds, he can tell approximately at what depth the coal should be found. Since the Pittsburg coal is used as a reference stratum from which the position of the oil and gas sands is calculated, it is particularly necessary to note its horizon.

#### ROCKS SHOWING AT THE SURFACE.

The surface rocks in this county belong entirely to the Carboniferous system. Four formations are represented, the Greene, Washington, and Monongahela being present entire and the upper 100 feet of the Conemaugh showing along the eastern edge of the county, where the Pittsburg coal rises above the level of Monongahela River. The Greene and Washington formations comprise the Dunkard group.<sup>a</sup>

#### GREENE FORMATION.

The section from the highest rocks exposed in the State down to the top of the Upper Washington limestone is included in the Greene formation, which is about 700 feet in maximum thickness and is composed largely of shale and shaly sandstone. These rocks are so deeply weathered on the hills that little can be determined of the details. The formation contains also some locally developed massive sandstone, red shales, small limestone beds, and two or three very thin coal seams.

<sup>a</sup>For more complete descriptions of the surface rocks the Masontown-Uniontown folio (No. 82), Waynesburg folio (No. 121), Amity folio (in preparation), and Rogersville folio (in preparation), *Geologic Atlas United States*, should be consulted.

## WASHINGTON FORMATION.

A series of comparatively soft rocks extending from the top of the Upper Washington limestone down to the roof of the Waynesburg coal comprise the Washington formation. The Upper Washington limestone is a conspicuous and rather persistent member of the Dunkard group, although it is absent in portions of the southern and western parts of the county. Its distance above the Waynesburg coal varies considerably on account of the variable thickness of the formation, but is between 300 and 400 feet.<sup>a</sup> Throughout much of the county this limestone weathers to a grayish white, a color so much lighter than that of other limestones that it is a distinct characteristic. The Upper Washington, where seen, is from 4 to 15 feet thick and is broken into two or more beds by thin layers of shale. On fresh fracture the rock is generally blue or black and mottled. The Lower Washington limestone is a bed in places 10 feet thick, which occurs a short distance above the Washington coal and 100 to 150 feet above the base of the formation.

Other beds of limestone up to 10 feet or more in thickness appear locally in the section. One of these, the Jollytown limestone, 30 feet above the coal of the same name and 20 feet below the top of the formation, is a persistent little bed, coarsely brecciated, weathering to a dull gray, in many exposures tinged with yellow.

A number of coal beds occur in the formation, but only one of them is of any importance. This is the Washington coal, which is opened below the Lower Washington limestone at a few points along the valleys of Dunkard Fork and Crabapple Creek, in Richhill Township. The other coals are too thin and too much broken up by partings to be worked until the thicker and more valuable beds in the Monongahela formation below are exhausted. The names Waynesburg A, Waynesburg B, and Jollytown are applied to them. Overlying the Waynesburg coal is the Waynesburg sandstone, which when massive is 40 to 70 feet thick.

## MONONGAHELA FORMATION.

This formation extends from the top of the Waynesburg coal to the base of the Pittsburg coal and in Greene County is from 273 to 405 feet thick. The whole thickness is exposed in the county only at the mouth of Tenmile Creek and on Monongahela River above Grays Landing.

The Monongahela formation contains over 100 feet of limestone, some heavy beds of sandstone, shales, and five coal seams which are more or less persistent. The Waynesburg coal is from 4 to 6 feet thick. It outcrops extensively in the eastern part of the county

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<sup>a</sup> Intervals given thruout this paper are from top to top of the respective beds.

and is also exposed on two branches of Wheeling Creek, on the western border. It is reported in a large number of wells.

A stratum of coal a foot thick, known as the Uniontown, lies about 90 feet below the top of the formation, being separated from the Waynesburg coal by a sandy shale and the Waynesburg limestone.

Below the Uniontown coal is the Benwood limestone, occupying about two-thirds of the 150-foot interval to the Sewickley coal. Shale and sandstone beds break up the Benwood into two or more divisions, each of which is made up of layers of white, gray, and drab limestone a foot or more thick. The Benwood limestone outcrops on Meadow Run below Davistown and in the valley of Tenmile Creek below Jefferson. Particularly good exposures occur on Castile Run and at the iron bridge across Tenmile Creek, 2 miles below Jefferson.

Another persistent coal bed in the Monongahela formation is known as the Sewickley. It is 100 to 120 feet above the Pittsburg coal and varies in thickness from 20 inches to 4 feet or more. It outcrops only in the main stream valleys on the eastern side of the county, and it has been mined extensively for local use at Mapletown. To most well drillers it is known as the Mapletown coal.

The Redstone coal, from 40 to 70 feet above the Pittsburg bed, is seen in occasional outcrops below Clarksville, where it shows about 6 inches of coal and bituminous shale. This bed probably thickens in the southeastern part of the county, because 3 miles south of Dunkard, on Robinson Run, in West Virginia, it is 4 to 5 feet thick and 40 feet above the Pittsburg seam.

The Pittsburg coal, at the base of the formation, outcrops for a short distance in the northeast corner of the county. Near the mouth of Tenmile Creek it dips below water level, but it reappears near the mouth of Whiteley Creek and continues above water level to the West Virginia line. It is from 6 to 10 feet thick and is a high-grade bituminous coal. Little is known regarding the character of the coal or the detailed section of its bed in the deeply buried parts of the basin in Greene County. Drill records, with few exceptions, attest its presence in all parts of the county, with about the same aggregate thickness as it maintains in the outcrop on the edges of the field at the surface and at depths ranging to more than 1,300 feet. A heavy bed of sandstone generally lies above the coal.

#### ROCKS BELOW THE SURFACE.

##### SOURCE OF DATA.

The present discussion of the rocks which lie below the Pittsburg coal is based entirely on the records of wells scattered throughout the county. In comparing the records of wells in any given township



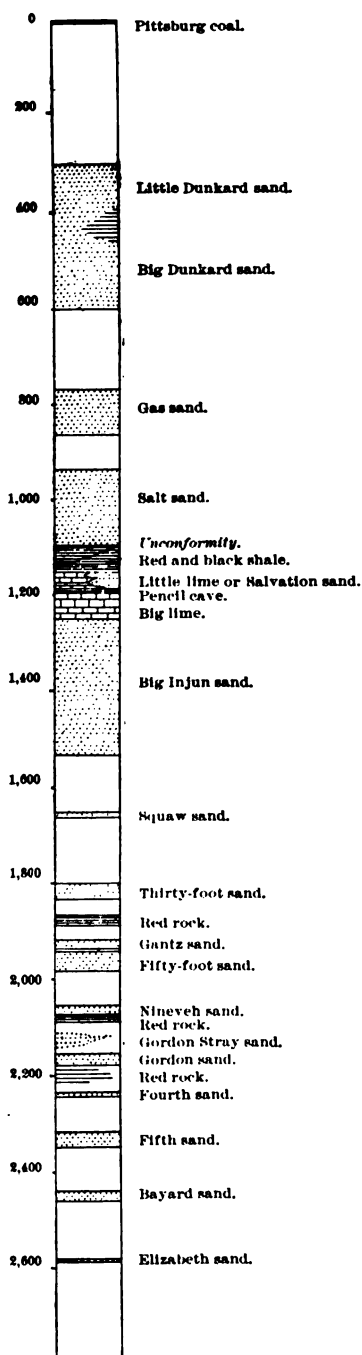


FIG. 1.—Generalized section of rocks below the Pittsburgh coal.

considerable variations are found in the stratigraphy as observed by competent drillers. This must necessarily be the case, for it is well known that the beds have a limited geographic development; some thicken in one direction and entirely disappear in another. Some of the most massive sands change to sandy shale or become careous in short distances.

The apparent variations in the stratigraphy shown by comparing the records of wells in close proximity to each other may be accounted for by the supposition that the drillers did not measure to the same bed, or by inaccuracies of measurement due to the stretching of the manila cable.

Furthermore, although the depth to the producing sands is measured by steel tape, the other depths are generally obtained by counting the turns of cable on the bull-wheel shaft, errors in this sort of measurement being easily made. The difficulty of identifying rocks by sand pumpings and the relative ease of drilling is a source of variation or error. All facts must all be considered in comparing the records of deep wells.

Details regarding position, thickness, and productiveness of the beds will be found on pages 30–44. The reader is referred to the general section (fig. 1) for a graphic representation of the sequence of the beds described below. This section shows only the approximate position and relation of the sands and omits details of character and distribution.

#### CONEMAUGH FORMATION.

Many of the records of wells drilled in Greene County give no detail between the Pittsburgh coal and Dunkard or first productive sand.

It is known, however, that limestone is encountered about 50 feet below the coal and that this is underlain by a massive sandstone the Connellsville, which in some places is 50 feet thick. Another heavy sandstone, the Morgantown, is found at a distance of 150 to 200 feet below the coal. The interval between these two sandstones is occupied by shaly sandstone or red shale. These beds of red shale in the upper part of the Conemaugh are probably continuous throughout the county, but in some sections the records are so incomplete that this can not be positively asserted. In the western part of the county most of the complete records note from 50 to 200 feet of red rock, and in Richhill Township it is reported even in the fragmentary records. In the eastern half of the county, however, red rock is not reported so uniformly. Several well records from Franklin Township indicate the presence of a considerable thickness of red beds. In the Waynesburg gas well (201),<sup>a</sup> which was drilled at the mouth of Purman Run in 1885,<sup>b</sup> red rock is reported as occupying 298 feet in an interval of 343 feet at this horizon. It is noted in the Fonner field and in the vicinity of Nineveh, in Morris Township, 100 to 250 feet below the Pittsburg coal. It is also found in eastern Washington County and is noted in many of the wells in Monongalia and Marion counties, W. Va. In the log of the Brice Wallace well, in Marion County,<sup>c</sup> red rock is recorded as extending through a space of nearly 300 feet.

The Morgantown sandstone, which lies beneath these red beds, is frequently reported in records of wells drilled in the western part of the county and is known there as the Murphy sand. In many places the shale underlying the Morgantown sandstone is red.

The Conemaugh formation in Greene County is from 570 to 670 feet thick. The lower half is composed largely of massive sandstone and sandy shale. This part of the formation is variable, being recorded in some logs as a continuous mass of sandstone and in others as two or more beds separated by shale. No two records are exactly alike in this respect. Where these rocks come to the surface there are generally two sandstones in this portion of the formation. They are known as the Saltsburg and Mahoning sandstones. Both of them are in some places almost continuous masses of heavy sand rock as much as 150 feet thick, with few or no breaks, while in other places either one or both become shaly. The upper portion of this sandy mass, lying 300 to 420 feet below the Pittsburg coal, is called the Little Dunkard sand and is considered equivalent to the Saltsburg sandstone, and the lower portion, known as the Big Dunkard sand, lying generally from 420 to 600 feet below the Pittsburg coal, is considered

<sup>a</sup> Numbers in parentheses refer to the same number at a well location on the county map (Pl. I). Logs of wells Nos. 1-170 are given in the table on pages 36-91.

<sup>b</sup> Carl, J. F., Ann. Rept. Geol. Survey Pennsylvania, 1886, Pl. II, p. 772.

<sup>c</sup> West Virginia Geol. Survey, vol. 1 (a), 1904, p. 238.

equivalent to the Mahoning sandstone. In several instances in various parts of the county sandstones which occur more than 600 feet below the Pittsburgh coal have been called Dunkard by the drillers. Where these are thin and where their bases do not extend more than 650 to 670 feet below the Pittsburgh, they may have been correctly named, but when their bases are lower than this it is probable that they are more nearly identical with the Upper Freeport sandstone, which lies below the Upper Freeport coal at the top of the Allegheny formation.

#### ALLEGHENY FORMATION.

In this region drillers rarely record any coal beds below the Pittsburgh. Having reached this well-known stratum, they look only for productive sands and often record nothing else, or hundreds of feet of rocks are lumped together as "shale" or "shaly sandstone." In the record of the Sayers well (202), which was drilled on the site of the pump station 2 miles east of Waynesburg, the one item "black shale, 370 feet," covers the entire Allegheny and Pottsville formations. The Johnstown and Vanport ("Ferroferous") limestones are noted in one or two records, but as a whole the data in this formation are meager and unreliable.

The Allegheny formation, extending downward from the top of the Upper Freeport ("Connellsville") coal to the top of the Pottsville formation, varies in thickness from 270 to 370 feet, though in the southwestern part of the county its thickness may be placed as low as 240 feet. Nine records in the western part of the county, which give the Upper Freeport coal and Salt sand, make the greatest interval 367 feet, the least interval 241 feet, and the average 282 feet. Although the Upper Freeport coal is not reported in many wells, it is believed to be fairly continuous. In the Allegheny Valley at least five workable coal beds occur in this formation, but the number of these extending underneath this corner of the State is unknown. In several wells, of which the William Milliken No. 2 (27), in Center Township, is an example, a coal bed is recorded 150 feet below the Upper Freeport. This is probably the representative of one of the Kittanning beds. In the J. B. Fordyce record (66), Gilmore Township, the name Connellsville is applied to a bed of coal 5 feet thick which is found 725 feet below the Pittsburgh. At an interval of 44 feet above this, directly underneath the Big Dunkard sand, lies a 3-foot bed of coal. It seems probable that this upper seam may be the true Upper Freeport, or "Connellsville," bed, in which case the lower seam would be the Lower Freeport coal. The lower coal lies only 197 feet above the Salt sand. There may be other reported occurrences of the "Connellsville" coal which are in reality the Lower Freeport instead of the Upper Freeport bed, in which case the Allegheny formation is locally thinner than *estimated above*.

The sandstones of the Allegheny are variable and may be found almost anywhere in the formation. On the surface they are known variously as the Upper Freeport, Freeport, Kittanning, and Clarion sandstones, according to their stratigraphic positions. Only one of the beds is recognized by the drillers in this region and that is called the Gas sand. Its top lies from 650 to 850 feet below the Pittsburgh coal, and its recorded thickness varies from 15 to 140 feet. This sand is not supposed by the writers to be a definite bed, but the name seems to be applied to any sandstone occurring between the Big Dunkard and Salt sands. In general, the Gas sand lies about the horizon of the Kittanning or Clarion sandstone, near the middle or base of the formation, though in some records it seems to be placed still lower, in the subjacent Pottsville.

#### POTTSVILLE FORMATION.

The thickness of the Pottsville formation varies between 70 and 200 feet, being as a rule greatest in the eastern part of the county. In general, the formation consists of two beds of heavy sandstone, separated by shale. The upper is the Homewood and the lower the Connoquenessing sandstone of the Beaver Valley region. Many well records show these sandstones merged into one continuous bed.

In eastern Greene County a sand rock reported at an average depth of 765 feet below the Pittsburgh coal and commonly known as the Gas sand, seems in some cases to be equivalent to the Homewood sandstone. The recorded thickness ranges from 15 to 140 feet. In western Greene County, as noted above, the term Gas sand is applied to a bed of sandstone in the Allegheny formation.

The top of the Salt sand occurs at an average distance of 930 feet below the Pittsburgh coal. Its thickness varies from 15 to 200 feet. Where the Gas sand is equivalent to the Homewood sandstone, the Salt sand corresponds with the Connoquenessing sandstone. Where the Gas sand is absent or not noted, or in the Allegheny, and the Salt sand is only moderately thick, the latter may be equivalent to either the Homewood or Connoquenessing sandstone. When the Salt sand is recorded as being extremely thick, however, it usually coincides with the whole Pottsville formation and may include the upper part of the Mauch Chunk.

In general, the Pottsville formation is supposed to correspond in its limits with the Salt sand, but it is probable that locally in eastern Greene County the upper portion of the formation includes the Gas sand. In places a bed of dark shale occurs below the Salt sand and above the red shale of the Mauch Chunk formation. It can not be stated positively whether this belongs to the Mauch Chunk or Pottsville.

## MAUCH CHUNK FORMATION.

The Mauch Chunk formation consists generally of a mass of red shale and underlying limestone, with here and there a sandstone bed between them. In some places the upper part of the formation consists of shale which is not red. This is especially true in the western part of the county, where red rock is rarely reported in this formation. To the east, however, it is more abundant, and in the vicinity of Waynesburg attains a thickness of over 100 feet. The Mauch Chunk formation increases in thickness, from 100 feet in the northwest corner of the county to over 250 feet in Monongalia and Marion counties, W. Va. The lessened thickness toward the north and west is due to an unconformity, caused by a period of elevation and erosion after the deposition of the Mauch Chunk and before the overlying Pottsville beds were laid down.

Throughout the greater portion of the county a bed of limestone 20 to 70 feet thick, known to drillers as the Little lime, occurs below the shaly portion of the Mauch Chunk. Toward the southwest, especially in Springhill and portions of adjacent townships, sandstone is reported at this horizon, to which the term Salvation sand is commonly applied in Pennsylvania and Maxton sand in West Virginia. Below the Little lime lies a few feet of soft shale that breaks up into small splinters in which the drill sinks rapidly. Owing to the peculiar way in which it breaks and its tendency to cave, this bed is known as the "Pencil cave."

The portion of the formation below the Pencil cave consists entirely of limestone, and is known as the Big lime. Sometimes the Little lime is not reported and the Pencil cave may be absent, in which case the entire thickness of limestone is reported as Big lime. These limestones are known over considerable areas as the Mountain limestone and correspond to the Greenbrier limestone which outcrops on Chestnut Ridge and Laurel Hill. In Greene County limestone seems to be everywhere present in the Mauch Chunk formation and is a good datum for the drillers.

## POCONO FORMATION.

The top of the Pocono formation is coincident with the top of the Big Injun sand of the well drillers, which corresponds with the Burgoon sandstone of the Allegheny Front. The thickness of the formation varies from 300 to 900 feet, according to the position of its base as interpreted by different authorities. Considerable doubt exists regarding this point, as there is a strong resemblance between the Pocono rocks and those of the Chemung formation at the top of the Devonian, and even where they outcrop it is difficult in many places to draw any definite line of separation between them.

In his report on the geology of West Virginia,<sup>a</sup> I. C. White gives the Pocono a thickness of 560 to 600 feet, and includes in it the Big Injun and Squaw sands, with 380 feet of underlying shales and sandy beds. The Big Injun in White's section is 150 feet thick. He classes as Catskill a series of sands and shales 571 feet thick, extending from the bottom of the Pocono, which he places just above the Gantz sand, to the bottom of the Elizabeth sand, and places in the Chemung the gray and dark shales, with an occasional shell, which lie below the Elizabeth sand. On the other hand, J. J. Stevenson<sup>b</sup> states that he found along the National pike in Fayette County, within 18 inches of the base of the Pocono sandstone, a number of species, which, according to James Hall, are typical Chemung forms. The position of these fossils clearly shows that from a paleontologic standpoint no formation can be present in that region between the Pocono sandstone and the Chemung shale. The Pocono in this locality is approximately 300 feet thick and composed almost entirely of sandstone which varies from thin-bedded flaggy rock to massive conglomerate. Following Stevenson, M. R. Campbell<sup>c</sup> describes the Pocono as being about 400 feet thick and equivalent to the Big Injun sand of the drillers. He classes the sands and shales below the Big Injun as Devonian. The presence of so-called Chemung fossils, however, does not preclude the possibility that the rocks containing them are Pocono in age, as the fauna may represent a merging of Devonian into Carboniferous forms. That there may be such a zone of merging with no distinct boundary is shown by Charles Butts in a paper on the Olean quadrangle in southern New York.<sup>d</sup> He finds that a few distinctively Devonian forms persist and are associated with forms which belong in the Mississippian and with other forms which have Carboniferous aspects. Carboniferous fossils have not been found below the Burgoon sandstone in the Chestnut Ridge region, and, although thorough search has not been made, it seems probable that they are not there.

In the Latrobe and other quadrangles in that vicinity a bed of red shale has been recognized from 350 to 500 feet below the top of the Big Injun sand and has been correlated with a bed of red shale which outcrops at Patton, Jefferson County. This shale, known as the Patton shale, carries fossil plants which have been identified by David White as Pocono forms. The Pocono formation in that region, therefore, is considered to be at least 400 to 500 feet thick.

With the exception of the exposure on the National pike, the Pocono outcrop nearest to Greene County of which a measurement has been

<sup>a</sup> West Virginia Geol. Survey, vol. 1, 1899, p. 205.

<sup>b</sup> The upper Devonian rocks of southwest Pennsylvania: Am. Jour. Sci., 3d ser., vol. 15, 1878, pp. 423-426.

<sup>c</sup> Masontown-Uniountown folio (No. 82), Brownsville-Connellsville folio (No. 94), and Latrobe folio (No. 110), Geologic Atlas U. S.

<sup>d</sup> Fossil faunas of the Olean quadrangle: Rept. New York State Paleont., 1902 (Bull. N. Y. State Mus. No. 39), pp. 290-295.

made is along the Pennsylvania Railroad east of Bennington, Blair County, on the Allegheny Front. This was measured by Charles Butts,<sup>a</sup> who collected fossils from this locality and came to the conclusion that although its base is rather indefinite the Pocono formation there is about 1,000 feet thick. In the Kittanning folio (No. 115) Butts describes this section in considerable detail and correlates it with well sections at Johnstown and in Indiana and Armstrong counties. A mass of red shales which he finds 1,000 feet below the top of the Burgoon sandstone is recognized as probably the Catskill formation at top of the Devonian, and these shales and the beds between them and the top of the Burgoon have been traced by means of various well sections into the Kittanning quadrangle; but the wells are at such a distance from each other that the identification can not be regarded as positive. The red beds of the Kittanning region lie not far below the Hundred-foot (Gantz and Fifty-foot) sand. From this Butts concludes that the Hundred-foot, Big Injun, and intervening sands are included in the Pocono and that the boundary between the Carboniferous and Devonian systems in Armstrong County should be considered as the top of the first red rock below the Hundred-foot sand. L. H. Woolsey has compared a large number of well sections and from them traced the horizon of the Hundred-foot sand and underlying red shale (Catskill) across Butler County to the Beaver quadrangle. A similar study was made by F. G. Clapp in work on the Amity quadrangle, in eastern Washington County. A great number of well records in western Pennsylvania were compared, and the beds between the Beaver and Amity quadrangles were correlated. There seems to be little question that the Hundred-foot sand of the northern counties is equivalent to the Gantz and Fifty-foot at Washington, and the tracing from Washington into Greene County is equally certain. In the same way the Berea sandstone of Ohio has been traced into this region, by means of well records, through Jefferson County, Ohio, Brooke County, W. Va., and Washington County, Pa., and it has been found to occur near the same horizon as the Thirty-foot sand. Throughout Greene County this sand lies from 20 to 80 feet above a certain bed of red shale, which is useful in correlating and which is supposed to be equivalent to the Bedford red shale of Ohio. Another group of red shale beds, similar to those called Catskill or sub-Blairsville in the Latrobe quadrangle, occurs in Greene County in all the wells which have penetrated that horizon.

Thus in tracing the formations into Greene County from the east and from the north and west two dissimilar conclusions have been reached. When traced from the east there seems to be little question that the Pocono formation is equivalent to the Big Injun sand only. When traced from the north and west, it is concluded that

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<sup>a</sup> Ebensburg folio (No. 133), Geologic Atlas U. S.

the formation must be about three times as thick, including the Thirty-foot, Gantz, and Fifty-foot sands. For this reason it is necessary to leave the base of the Pocono in doubt, although in this report it is provisionally placed at the top of the Nineveh sand, which is supposed to be the uppermost bed of the Catskill formation.

The Big Injun or Mountain sand lies immediately below the Greenbrier limestone, or Big lime, of the Mauch Chunk formation, and its top is therefore everywhere definite, making a good reference plane in drilling. The sandstone constitutes the upper part of the Pocono formation and on the surface is known as the Burgoon sandstone. In Greene County it attains a thickness of 200 to 300 feet. In places it contains a break of shale up to 50 feet thick. The interval from the Pittsburg coal to this sand varies from 1,073 feet in the David Stickler well, near Simpson Store, to 1,345 feet in the Mary McClure well, in Perry Township.

Generally there is an interval of 250 to 350 feet below the Big Injun sand occupied by "slate and shells." In a few records, however, the Squaw sand is reported 50 to 150 feet below the base of the Big Injun. This sand locally amounts to 100 feet in thickness, although it is generally less than 50 feet. In Washington County it is a rather prominent bed.

The lower portion of the Pocono formation is composed of sandstone beds separated by shales. These sandstones in one place or another carry gas or oil. They occur in the following order, from the top down: Thirty-foot sand, Gantz sand, and Fifty-foot sand.

#### DEVONIAN SYSTEM.

About 2,000 feet below the Pittsburg coal occur the uppermost rocks supposed to be of Devonian age. They underlie the Pocono formation and consist of sandstones and shales of varying thickness and composition. The best-known members are the Nineveh, Gordon, Fourth, Fifth, Bayard, and Elizabeth sands. In many places red beds occur in the upper part of this series. The red color is found in both sandstone and shale, and these beds are believed to be the feather-edge of the Catskill formation of eastern New York and Pennsylvania.

In Greene County the Catskill may be placed either at the top of the Devonian or a few hundred feet below, its position in the stratigraphic column depending on the definition of the base of the Pocono. In the eastern part of the county the beds are several in number, attaining a total thickness of 100 feet or more, but in the western part they consist only of one or two thin beds which in few places exceed 30 feet in thickness. The uppermost of these is either the Nineveh sand or a bed directly underneath it. This red stratum is an excellent horizon marker. The other principal bed



of this series in western Greene County is less persistent, but where present it lies 75 to 100 feet below the bed just described, between the Gordon and Fourth sands. In wells where these beds are present, therefore, the Nineveh and Gordon sands can be almost certainly identified.

The Catskill formation should not be confused with the Bedford red shale, which occurs considerably higher, between the Thirty-foot and Fifty-foot sands. The Catskill formation increases in thickness toward the east, while the Bedford disappears in that direction and becomes more prominent toward the northwest. The Catskill is without doubt Devonian, and the Bedford, where it outcrops in Ohio, is of Carboniferous age.

The thickness of the Devonian rocks is not known. A well in Aleppo Township penetrated to a depth of 4,722 feet below the Pittsburgh coal, or about 2,700 feet into the Devonian. Two thousand feet of strata below the Bayard sand are reported in the record of this well as "shells and slate," meaning thin beds of shaly sandstone and shale.

#### WELL SECTIONS.

In Pl. II the sequence of the rocks in 21 deep wells is represented graphically. Nineteen of these wells are in Greene County, one at Washington, 20 miles north of Waynesburg, and one is at Leipsic, 13 miles north of Waynesburg. They are arranged in an east-west line from Monongahela River to Morris Township, and then irregularly southwestward to the corner of the State. Besides showing the correlation of sands throughout the county, these sections also show the correlation of the Nineveh and Gordon sands into Washington County, a subject about which there has been a difference of opinion and which is discussed on pages 44-45. The sections are aligned on the Pittsburgh coal. The correlation lines show the gradual diminution in thickness and final disappearance of the Mauch Chunk red shale in the northern and western parts of the county as the result of the unconformity at the base of the Salt and Waterford. The positions of the various oil and gas sands are shown and their relations are indicated.

#### GEOLOGIC STRUCTURE.

##### INTRODUCTION.

Greene County is near the center of the great bituminous coal basin. The structural features have the general northeast-southwest trend which is characteristic of most of the Appalachian province, while the crests of the anticlines and the troughs of the synclines crossing this county are successively lower from east to west. These folds in the rocks are shown by the contour lines (or lines of equal elevation) on the accompanying map (Pl. I). The floor of

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**Ben Williams,  
Greensboro.**

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g coal is the reference surface, and at all points along a given line the Pittsburg coal has the same elevation above sea and on the adjacent contour the coal is 50 feet higher or lower, as may be. Thus the contour lines represent the shape of ls. The Pittsburg coal is seen at the surface in Greene only at the mouth of Tenmile Creek and on Monongahela above Grays Landing. In other parts of the county its position has been calculated from higher beds, which show at the surface from the records of a large number of deep wells. Enough data have been obtained to make the determination of the position of the coal accurate within a contour interval, although the bed is about a hundred feet below the surface throughout much of the county. The geologic structure is described from east to west across the county.

#### FAYETTE ANTICLINE.

The arch in the rocks is strongly developed across Fayette and Ireland counties. The axis crosses Redstone Creek above Pittsburg and approaches Monongahela River near the State line, in Greene County, if at all, in the extreme southeast corner. Although the axis may not cross this county, the western flank of the anticline is shown in the dip of the rocks from Dilliner to Mount Morris.

The evidence for determining the structure in this corner of the county is the outcrop of the Pittsburg coal along Dunkard and its depth in wells drilled between Mount Morris and Mount Pleasant. The crest of the fold as it crosses Fayette County is in elevation toward the south, and at the Greene County line the Pittsburg coal is about 1,150 feet above tide. From the Mount Morris the dip of the beds down the flank is fairly uniform and carries the coal down to 500 feet above tide.

#### LAMBERT SYNCLINE.

The syncline next on the west of the Fayette anticline has an irregular shape and its axis pursues an indirect course, crossing Monongahela above Browns Ferry and entering Greene County with a westward trend. A mile west of the river it turns southward, passing through Paisley and terminating somewhat indefinitely near Willow. The axis rises to the south, so that the Pittsburg coal, which is at about sea level at the river, is 150 feet higher at the south end of the basin.

#### BROWNSVILLE ANTICLINE.

The rocks rise westward from the Lambert syncline in an irregular arch which is called the Brownsville anticline. The axis of this fold is just east of the town of Brownsville, crosses Monongahela at East Riverside, and pursues a southerly course to Turkey. In Greene County this anticline is but a slight undulation.

and poorly defined, but it has the effect of throwing the strike of the flank of the Fayette anticline in the vicinity of Davistown into a north-south direction. The Brownsville anticline is a minor wrinkle in a large synclinal basin, the eastern limb of which rises to the Fayette anticline and the western limb to the Bellevernon anticline, which lies farther west. The determination of structure here is from the outcrop of the Waynesburg coal.

#### WHITELEY SYNCLINE.

The Whiteley syncline lies between the Fayette and Bellevernon anticlines, as does the Port Royal syncline farther north. The Port Royal syncline, however, loses its basin-like character in the vicinity of Fayette City, on Monongahela River, and the axis does not seem to be continuous with the axis here described.

Conditions are such that the accurate determination of the structure of this basin is almost impossible. The rocks showing at the surface are of such a character that tracing and definite correlation are impracticable, and in the 60 square miles of the basin lying between Muddy Creek and the State line there are only seven deep wells which give any light on the depth of the Pittsburg coal below the surface. Therefore the location of the structure contours in this part of the area is regarded as approximate only and subject to revision when definite evidence is available.

The Whiteley syncline is broad and shallow, deepening toward the south. The axis of the trough probably corresponds with a line through Fordyce and Kirby and crosses Dunkard Creek just south of the State line, about a mile west of Pentress, W. Va.

#### BELLEVERNON ANTICLINE.

The term Waynesburg anticline was applied to this fold in the rock structure by J. J. Stevenson,<sup>a</sup> who also applied the same name to the syncline on the west. The first work of the United States Geological Survey on this anticline was in Washington and Westmoreland counties, where it attains its maximum development at Bellevernon, on Monongahela River, and the anticline was named from that place, there being some doubt at the time as to its continuity with the axis supposed to exist at Waynesburg and also as to the applicability of the term Waynesburg to both the anticline and syncline lying next to the west. Later the survey of the Waynesburg quadrangle proved the continuity of the fold, but its location is so far east of the village of Waynesburg and it has in that area lost so much of its prominence that the term Waynesburg seems more appropriate for the syncline, and the name Bellevernon is retained for the anticline.

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<sup>a</sup>Second Geol. Survey Pennsylvania, Report K, 1876.

The Belleverson anticline is the most pronounced and important structural feature in Greene County. Its axis crosses Tenmile Creek and the county line a mile northeast of Clarksville and holds a direct southwest course for 14 miles to the head of Smith Creek, where it turns to the south between Roberts and Rudolph runs and gradually flattens out.

A normal continuation of the axis would cross the State line nearly a mile west of Blacksville, but for 2 miles west of that village the rocks are horizontal or rise to the east. It seems probable that the anticline disappears soon after entering West Virginia, if it crosses the State line at all. A low anticline which is seen just east of Blacksville probably has no connection with this fold, but is the north end of another axis.

The Pittsburg coal, which is the reference stratum on which the structure contour lines are drawn, is at an elevation of 1,000 feet at the point where the Belleverson anticline crosses Monongahela River between Belleverson and Charleroi; it is 750 feet above tide at Clarksville and only 400 feet above tide at Blacksville. It should be noted that conditions are favorable for the accurate determination of the position of the reference stratum along the Belleverson anticline in Greene County. The Waynesburg coal outcrops on Tenmile Creek from Monongahela River to Waynesburg, and the interval between it and the Pittsburg coal is known. Moreover, the depth of the Pittsburg coal below the surface is shown in 140 deep wells located along the anticline, and when the elevation of the well mouths is known the elevation of the reference stratum above mean sea level is easily determined. It is believed that the true position of the Pittsburg coal at any point on the west flank of the anticline down to the 400-foot contour line varies scarcely more than 10 feet from the position indicated on the map.

The eastern slope of the Belleverson anticline is short and gentle in the region of the greatest development of the Waynesburg gas field. From the crest of the fold near the mouth of Braden Run on Tenmile Creek to the bottom of the Whiteley syncline on Muddy Creek is a fall of scarcely more than 200 feet. From the same point to the axis of the Waynesburg syncline on the west the Pittsburg coal descends fully 400 feet. The regularity of the western flank of the Belleverson anticline is shown by the contour lines on the map.

#### WAYNESBURG SYNCLINE.

The structural basin which lies west of the Belleverson anticline was called the Waynesburg syncline by Stevenson.<sup>a</sup> He located the axis somewhat farther east than is shown on the accompanying map, but the basin is essentially the same as that described by him, and

<sup>a</sup> Second Geol. Survey Pennsylvania, Rept. K, 1876.

hence the name is retained. The axis is continuous with that of the Pigeon Creek syncline, described in the Brownsville-Connellsville folio (No. 94). When the Brownsville quadrangle was surveyed it was recognized that the Pigeon Creek and Waynesburg synclines might be one and the same, but mine data in the vicinity of Bentleyville, Washington County, seemed to indicate cross structure, and so the basin was given the local name of Pigeon Creek. Subsequent survey of the Amity quadrangle proved that these synclines are continuous, and consequently the name Pigeon Creek is dropped. On Pigeon Creek, Washington County, the Pittsburg coal is 700 feet above tide, while on Wisecarver Run, Greene County, it is at least 400 feet lower.

The axis of the Waynesburg syncline crosses the Washington-Greene county line about a mile west of Castile, Ruff Creek near the mouth of Boyd Run, and Browns Creek at Rees Mill, 2 miles west of Waynesburg. Continuing in a southwesterly direction it intersects South Fork of Tenmile Creek one-half mile above Pursley Creek, then swings southward, strikes Wayne Township near the head of Pursley Creek, turns slightly southwestward, and passes just west of the village of Hoovers Run. It crosses Pennsylvania Fork of Dunkard Creek 1 mile east of Jollytown.

The structure of this basin is determined by the records of several deep wells which show the position of the Pittsburg coal, and in the northern part of the county by the elevation at the surface of the Upper Washington limestone. It is possible that the basin may be somewhat deeper in the vicinity of Waynesburg than represented on the map. A well on the Wisecarver farm, on Wisecarver Run, found the Pittsburg coal about 285 feet above sea level, but the evidence was not sufficient to warrant drawing the 300-foot contour line on the coal. Near the head of Pursley Creek the coal lies about 350 feet above tide, and on the southern boundary of the State its elevation is about 300 feet.

#### AMITY ANTICLINE.

From the trough of the Waynesburg syncline the rocks rise to the west more gradually than to the east, and a low anticlinal fold enters the county in the northwest corner of Washington Township. This fold has been traced northward into Washington County beyond the village of Amity and is named from that place. Stevenson and White called it the Pinhook anticline, from a locality in Amwell Township, Washington County. The name was taken from a burlesque appellation of the little village of Lone Pine, and for that reason it is not suitable for geologic nomenclature. Where the fold enters Greene County from the north its crest is probably not more than 100 feet above the axis of the Waynesburg syncline, but it rises

rapidly in Washington County and becomes a more pronounced structural feature.

According to deep-well records the Pittsburg coal is about 475 feet above sea level on the crest of the Amity anticline at the point where it crosses the northern border of the county. One mile east of Hackneys, Washington County, where the axis crosses Tenmile Creek, its elevation is 495 feet. At Amity it is about 550 feet and at Lone Pine about 650 feet. In the vicinity of Swarts the Amity anticline turns directly southward, but at Browns Creek it veers slightly to the southwest and passes almost directly through the town of Rogersville, near which it seems to die out. At Rogersville the coal is probably less than 350 feet above sea level. West of the Amity anticline the rocks dip with a medium grade to the bottom of the Nineveh syncline.

By reference to the structure map (Pl. I) a dome-like arrangement of the contours is seen nearly in line with a continuation of the Amity anticline in the vicinity of Buzz, Bluff, and White Cottage, suggesting a continuation of the anticline in this part of the county. The structure is not definitely known here on account of a lack of well records, but from the evidence gathered in tracing local limestones and associated beds on the surface it seems to be about as represented on the map. The Pittsburg coal is about 400 feet above sea level just east of White Cottage and about the same altitude 1 to 2 miles southeast of Bluff. Over a considerable area between White Cottage and Pursley Creek the structure is rather flat. South of this area there is a faint continuation of the anticlinal arch to the State line, but the rocks are descending, the coal being 342 feet above sea in a well on Blockhouse Run and 331 feet on Garrison Fork of Dunkard Creek. The arch is here barely 50 feet above the bottom of the adjacent syncline on the east.

#### NINEVEH SYNCLINE.

This is one of the most pronounced troughs in Greene County. It was recognized and named by Stevenson in 1876. It enters the county in Morris Township and takes a course averaging S. 40° W., but varies 25° from this course. It passes just west of Nineveh, 1 mile west of Rutan, 1 mile southeast of Bristoria, 1 mile northwest of Higbee, and nearly through Aleppo and Morford.

The Nineveh syncline is located about the center of the Appalachian basin, of which it is one of the deepest troughs. At two points, near Rutan and in the vicinity of Morford, the Pittsburg coal at the center of the basin is only about 100 feet above sea level. The Morford depression is very small and rather hypothetical, but the Rutan basin contains one well in which the coal was found as low as 95 feet above sea level. The location of the axis of the Nineveh syncline is



better determined than any of the other axes in this part of the county, as numerous wells have been drilled on both sides of it for a great part of its course. Between Aleppo and Lick Run especially it is clearly defined by the wells of the Bristoria and the north end of the New Freeport oil fields. The mapping at Nineveh is likewise very close. At the northern edge of the county, between Rutan and Nineveh, and southwestward from Aleppo, however, it has been necessary to interpolate the coal contours entirely on the basis of surface rocks. With the exception of the two deep basins mentioned above, the coal in the bottom of the Nineveh syncline lies in general between 100 and 200 feet in elevation. North of Nineveh it is rising and on the edge of the county attains an altitude of about 300 feet.

East of the Nineveh axis the structure is rather diverse. In the vicinity of Nineveh there is a fairly uniform rise to the crest of the Amity anticline, but south of this place the anticline dies out and the dips grow more gentle. In western Gilmore and southwestern Jackson townships is an area of 15 to 20 square miles in which little can be determined of the structure, as rock exposures are poor and no wells have been drilled. From indications gathered along the eastern border of the New Freeport gas field and from the records of two wells in southern Gilmore Township there seems to be a slight depression here, like the north end of a syncline which may possibly develop to the south, but nothing is known of this basin outside of Greene County. Between New Freeport and Triumph, in the depression mentioned, the Pittsburg coal is less than 240 feet above sea level. The small anticline separating this minor basin from the Nineveh syncline is well marked, passing directly through New Freeport and thence continuing in a southwesterly direction. On the west side of the Nineveh axis from Bristoria southward to Fish Creek the positions of the 200- and 250-foot coal contours are in places well determined, owing to the great oil development in this locality. Northwest of Deep Valley and northwest of Morford the dips of the surface sandstones and limestones seem to indicate two small domes in the structure. In the southwest corner of the county the positions of the contours are fairly well determined, owing to a few scattering wells.

West of the Nineveh syncline throughout its entire length the rocks rise rapidly to the crest of the Washington anticline. The greatest rise is between Rutan and Jacksonville, and amounts to 600 feet.

## WASHINGTON ANTICLINE.

This fold is a prominent structural feature. Stevenson named it in 1876 from the town of Washington, near which it passes and from which it extends southwestward, entering Greene County in Richhill Township near Simpson Store. Thence it sweeps to the west in a fairly regular curve, passing through Jacksonville and just southeast of Ryerson Station, and thence into West Virginia with a course S.  $60^{\circ}$ - $70^{\circ}$  W. The elevation of the Pittsburg coal on the axis of this arch varies from 480 to 705 feet above tide.

The most prominent feature of the fold in this county is a roughly triangular dome, having its center at Jacksonville. North of this dome the axis plunges to the vicinity of Simpson Store, where the elevation of the coal is about 480 feet, but beyond this place it rises again.

The grade along the southeastern flank of the Washington anticline in general averages 100 feet per mile, but between Graysville and Bristoria it amounts to 250 feet per mile. In this region and as far north as Enslow Fork of Wheeling Creek the contours are rather accurately placed, being determined by logs of wells in the Richhill gas field.

The dips on the western flank of the Washington anticline are much more gentle. Information about the structure of this part of the county has been derived from outcrops of the Waynesburg and Washington coals on Crabapple Creek and Enslow Fork and from a few limestone and sandstone beds. There are a few scattering wells in this vicinity. The dip of the rocks rarely exceeds 100 feet per mile and in some places is less than 50 feet. West of Jacksonville a spur of the anticline shoots off to the northwest, reaching as far as the western edge of the county.

## OIL AND GAS SANDS.

## INTRODUCTION.

In western Pennsylvania all oil and gas yet discovered have been produced from beds of sandstone, or "sands," as they are called by the drillers. The beds penetrated have been given various designations from time to time, and these terms have gradually come into common usage as descriptive of the beds. In order to show their general relations the following table has been prepared, giving the drillers' terms, the corresponding geologic terms, and the formation to which the sands belong:

*Drillers' terms for oil and gas rocks, etc., and their geologic correlation.*

Formation.	Name applied by drillers.	Geologic name.	Approximate maximum thickness.	Correlation with sands in neighboring fields.
			<i>Feet.</i>	
Washington.....	Washington coal.....	Washington coal.....	3	
	Bluff sand.....	Waynesburg sandstone.	60	
Monongahela.....	Waynesburg coal.....	Waynesburg coal.....	5	
	Mapletown coal.....	Sewickley coal.....	8	
	Pittsburg coal.....	Pittsburg coal.....	10	
	Murphy sand.....	Morgantown sandstone.	30	
Conemaugh.....	Little Dunkard sand.	Saltsburg sandstone..	100	
	Big Dunkard or Hurry-up sand.	Mahoning sandstone..	150	
Allegheny.....	Upper Freeport or Connellsville coal.	Upper Freeport coal..	6?	
	Gas sand <sup>a</sup> .....	Kittanning, Clarion, or Homewood sandstone.	150	
Pottsville.....	Salt sand.....	Conoquenessing or Pottsville sandstone.	200?	
	Red rock or shale.....	Mauch Chunk shale...	150	
Mauch Chunk.....	Little lime (or Salvation sand).	Greenbrier limestone..	100	Maxton sand.
	Pencil cave.			
	Big lime.....			
Pocono.....	Big Injun sand.....	Burgoon sandstone...	300	Mountain sand.
	Thirty-foot sand.....		100	Berea sand.
	Gantz sand.....		40	First sand
	Fifty-foot sand.....		60	Hundred-foot sand.
	Nineveh or Nineveh thirty-foot sand.		40	
	Red rock.....		20	
Devonian.....	Gordon Stray sand.....		40	Gray, Bowlder, or Campbell's Run sand.
	Gordon sand.....		100	Third or Flat Run sand.
	Fourth sand.....		40	
	Fifth sand.....		40	McDonald sand.
	Bayard sand.....		12	Sixth sand.
	Elizabeth sand.....		10	

<sup>a</sup> When this name refers to the Kittanning or the Clarion sandstone it designates a part of the Allegheny formation.

Oil and gas are found in Greene County in paying quantities at several horizons. The oil sands are the Dunkard, Big Injun, Gantz, Fifty-foot, Nineveh, Gordon, and Fourth. The important gas sands include those just given and, in addition, the Salt, Fifth, Bayard, and Elizabeth sands. In using the driller's designations of the various beds, it is not supposed that all of them are in reality continuous

sandstone beds underlying the entire area. That is true of a few beds such as the Big Injun and Salt sand, but if the extent of the others is anything like that of sandstone beds covering large areas of the surface in western Pennsylvania, they are generally lentils in the formation rather than distinct beds by themselves. In order to show the occurrence of the various sands 21 deep-well sections are given in Pl. II and the correlations of the various beds are indicated.

Knowledge of the sands is necessarily fragmentary, but the records of a large number of wells in the territory have been obtained and compared, and from them considerable information has been compiled regarding the thickness, intervals, and productiveness of the various beds. As the Pittsburg coal, which underlies the entire region, is easily recognized and is used by drillers as a datum surface in measuring to the various beds, the position of the sands will be referred to it.

In order to give a general idea of the sequence of sands to be described, the following table is given, showing the maximum, minimum, and average distances from the Pittsburg coal to the top of the well-known sands.

*Distances from Pittsburg coal to gas sands in Greene County.*

Sand.	Maximum.	Minimum.	Average.	Sand.	Maximum.	Minimum.	Average.
	<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Murphy.....	232	170	211	Fifty-foot.....	2,044	1,902	1,937
Dunkard.....	575	360	478	Nineveh.....	2,121	1,997	2,051
Gas.....	856	674	765	Gordon.....	2,227	2,070	2,157
Salt.....	1,035	860	932	Fourth.....	2,298	2,140	2,230
Big Injun.....	1,345	1,073	1,221	Fifth.....	2,365	2,225	2,292
Thirty-foot.....	1,845	1,744	1,795	Bayard.....	2,464	2,388	2,433
Gantz.....	1,965	1,813	1,916				

## DESCRIPTION OF SANDS.

### MURPHY SAND.

In the western half of the county a sandstone called the Murphy sand is frequently reported by drillers. It generally occurs from 170 to 200 feet below the Pittsburg coal, but in Springhill Township the distance is as much as 220 feet. This sand runs from 5 to 30 feet in thickness and in exceptional cases reaches 70 feet. It corresponds approximately in stratigraphic position with a prominent surface sandstone known in southwestern Pennsylvania and northern West Virginia as the Morgantown sandstone, from the city of Morgantown, W. Va.

### DUNKARD SAND.

According to identifications by various drillers in different parts of the county, the Dunkard sand lies from 300 to 575 feet below the Pittsburg coal, and its thickness ranges from 40 to 150 feet. In the

eastern part of the county some drillers find a small break in this sandstone, and in the western part it consists of two quite distinct beds, the upper and lower divisions being known as the Little and Big Dunkard sands. The name is taken from Dunkard Creek, near the mouth of which some producing oil wells were struck in this sand about 1863. The wells at Willow Tree and in the Garrison oil field, 2 miles south of Willow Tree, are producing oil from the Dunkard sand. In other areas this sand is generally unproductive, although in several townships it has shown traces of oil or gas. In the western part of the county the Big Dunkard is frequently called the Hurry-up sand.

#### GAS SAND.

The Gas sand is not important, but several wells produce gas from it in the Richhill field, between Durbin and Owens Run. In some places it holds considerable salt water. The position of this sand varies, according to different well records, and it is possible that there are a number of sandstones at this horizon, any one of which may be called the Gas sand. The average depth below the Pittsburg coal is about 765 feet.

This sandstone may be regarded as the Homewood, the uppermost member of the Pottsville formation, or as the Clarion, at the base of the Allegheny formation. Its recorded thickness ranges from 15 to 140 feet, the variations being due in part to undoubted changes in the amount of sandstone present at this horizon and in part to a tendency on the part of the driller to class everything as sandstone for a considerable distance, when in reality it may be much broken by shale beds.

#### SALT SAND.

The Salt sand is so named because salt water is often encountered in drilling through it. In the Richhill field and in several wells on the crest of the small anticline south of Deep Valley it produces considerable gas, and smaller quantities are occasionally found in it elsewhere in the county.

This sand is about 930 feet below the Pittsburg coal, and, according to the records in hand, varies in thickness from 15 to 175 feet. It should be noted that in Greene County the Salt sand lies above the Big Injun sand, while in Armstrong County the same name is used for a gas-bearing stratum below the Big Injun.

Those records which give a thickness of over 100 feet for either the Gas or the Salt sand omit mention of the other, which shows that possibly both sands, being but little separated, have been included under one name.

## SALVATION SAND.

The Salvation sand is equivalent to the Maxton sand of West Virginia. It is reported only in Aleppo and Springhill townships, where it lies above the Big lime, separated from it only by the Pencil cave. Elsewhere in the county this horizon is represented by the Little lime. The sand is generally unproductive, but a number of wells in Springhill Township, on the small anticline south of Deep Valley, produce gas from it.

## BIG INJUN SAND.

The Big Injun sand rock is always recognized by the drillers from its thickness and position. It is usually from 250 to 300 feet thick, and, as shown by the records of over 500 wells scattered through the county, lies at an average of about 1,220 feet below the Pittsburg coal, ranging from 1,073 to 1,345 feet. In a general way the distance decreases northwestward from Dunkard and Perry townships to Richhill Township. In the following table the maximum, minimum, and average distances are given for the townships in the eastern half of Greene County:

*Distances from Pittsburg coal to Big Injun sand in eastern Greene County.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Perry.....	5	1,240	1,345	1,278
Dunkard.....	8	1,250	1,295	1,276
Wayne.....	56	1,217	1,316	1,268
Whiteley.....	6	1,230	1,305	1,257
Franklin.....	47	1,152	1,270	1,224
Jefferson.....	6	1,180	1,265	1,219
Washington.....	4	1,184	1,220	1,209
Morgan.....	7	1,185	1,220	1,205

Cumberland, Greene, and Monongahela townships are omitted from the list because of lack of evidence. The writers know of only 6 wells in Cumberland Township, 5 of which are dry. The record of only one of these, the Biddle well (31), at Carmichaels, is at hand, and shows that the Big Injun sand was found 1,241 feet below the Pittsburg coal and carries a small amount of gas. Many holes have been sunk in the eastern part of Greene Township, but almost all are shallow-sand wells. In Monongahela Township wells have been drilled at frequent intervals along Whiteley Creek, but most of them are shallow. A number of wells on Monongahela River begin below the Pittsburg coal, and of the others it is difficult to obtain records.

The intervals for the western half of Greene County are given by fields rather than by townships, because the records of neighboring groups of wells show considerable variation in the same township and

the amount of information available makes the finer discrimination of areas possible.

*Distances from Pittsburg coal to Big Injun sand in western Greene County.*

Field.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Fonner.....	9	1,158	1,235	1,183
Rogersville and Deertick.....	4	1,120	1,225	1,177
Nineveh.....	11	1,113	1,213	1,194
Grays Fork.....	5	1,147	1,215	1,174
Richhill.....	18	1,073	1,215	1,140
Wright Run.....	3	1,200	1,234	1,215
Sugar Grove.....	8	1,119	1,210	1,162
Alippo.....	13	1,215	1,248	1,227
Bristoria.....	124	1,180	1,280	1,221
Woodruff.....	7	1,203	1,263	1,236
Gilmor.....	3	1,275	1,283	1,280
New Freeport.....	162	1,171	1,265	1,234
Board Tree.....	5	1,240	1,269	1,250

It will be seen from the figures given in the tables that the interval decreases to the northwest. In other words, the average interval in

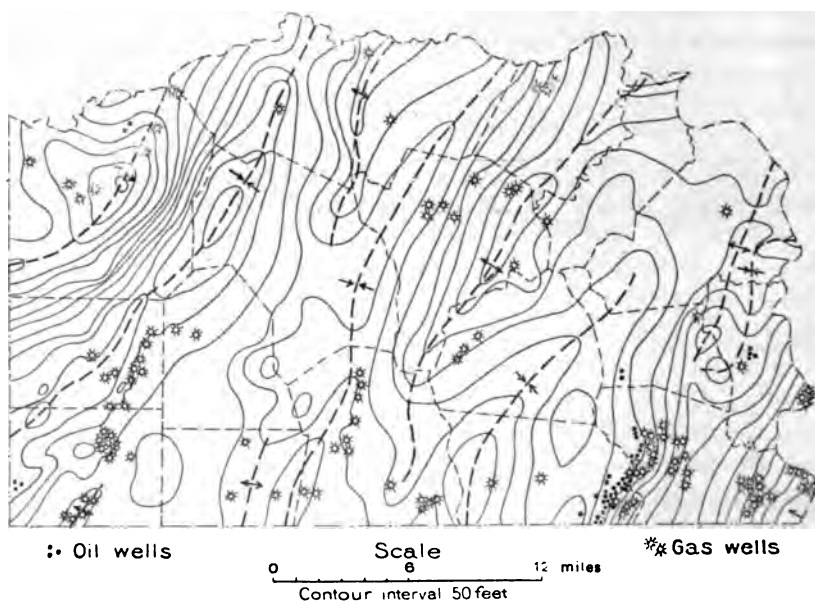


FIG. 2.—Structure map of oil and gas in Big Injun sand.

the southeast quarter of the county is over 1,270 feet and in the northwest quarter less than 1,200 feet. This decrease in distance between the Pittsburg coal and Big Injun sand is due to the thinning of the Mauch Chunk formation, as described in the section on stratigraphy (p. 18).

The Big Injun sand is generally expected to carry some gas. It does so in Morgan, Franklin, Richhill, and Dunkard townships, and in the New Freeport field. In the Hoovers Run field and in scattering

wells in Gilmore Township it ranks next to the Fifth sand as a gas producer. In the Mount Morris and Board Tree fields the Big Injun produces oil and, on the margins of some of the oil pools, gas.

The accompanying sketch map (fig. 2) of the geologic structure in Greene County shows the distribution of gas and oil in the Big Injun sand, so far as it can be determined from the records in hand. This map and those which follow (figs. 3-6) are necessarily somewhat incomplete, because many records fail to state which is the producing sand. Fig. 2 indicates the wide distribution of Big Injun gas throughout the county and shows particularly the structural relation of the Mount Morris oil pool.

#### SQUAW SAND.

An unproductive sand, usually less than 50 feet thick, is infrequently reported at distances varying from 50 to 150 feet below the base of the Big Injun. Wherever found it is known as the Squaw sand.

#### THIRTY-FOOT SAND.

The first sand commonly recognized below the Big Injun in Greene County is the Thirty-foot. Its distance below the Pittsburg coal varies from 1,729 to 1,882 feet. So far as known, it does not produce oil or gas anywhere in this county, nor can it be recognized by its thickness, as in that particular the well records show variations from 20 to 100 feet. In the Rebecca Hook (51) and John Miller (53) wells in the Waynesburg field the recorded thickness is 120 feet, but these records include in the Thirty-foot in one case the overlying shale and in the other probably the Gantz sand. In this county the Thirty-foot sand is not the same as the Thirty-foot in Butler and Armstrong counties, but probably corresponds with what is sometimes known there as the Gas, Butler, or Murrys ville sand. It agrees closely with the Berea sand of Beaver County, Pa., Jefferson County, Ohio, and Brooks County, W. Va. It is sometimes difficult to decide whether a sand noted as Thirty-foot in well records is in reality the Thirty-foot, Gantz, or some local sand, but this question can generally be answered by noting the positions of the red beds encountered by the drill. Between the Thirty-foot and Gantz sands lies a thin bed of red shale which is found in the wells of Washington County and generally in western Greene County, and which has been provisionally correlated with the Bedford shale of Ohio. The next lower red bed occurs 100 to 200 feet deeper, at the horizon of the Nineveh sand.

#### GANTZ SAND.

The Gantz sand is struck in the northern part of the county at distances varying from 1,870 to 1,946 feet below the Pittsburg coal. It takes its name from a well on the Gantz farm at Washington,



which was drilled in 1885 and was the first paying oil well in Washington County. The sand was found 1,827 feet below the Pittsburgh coal, the interval being less than in Greene County because of the disappearance of the Mauch Chunk shale toward the northwest. The Gantz sand is usually 10 to 25 feet thick. It produces oil and some gas in the Fonner field in Morris Township and is the principal gas sand in the vicinity of Clarksville.

#### FIFTY-FOOT SAND.

A producing sand which closely underlies the Gantz sand and in many places merges with it is known as the Fifty-foot. Here and there the interval between them amounts to as much as 40 feet, but in the western half of the county the two sands run together and in that region are nearly always reported as the Fifty-foot. Frequently where the two sands are separated only by a thin shale they are recorded as continuous. These sands correspond in horizon with the Hundred-foot sand in Butler, Armstrong, Beaver, and other counties.

The distance of the Fifty-foot below the Pittsburgh coal in the western half of the county varies from 1,900 to 2,034 feet. The figures for the various townships are given in the following table:

*Distances from Pittsburgh coal to Fifty-foot sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Richhill.....	35	1,900	1,992	1,955
Morris.....	16	1,905	1,966	1,944
Washington.....	4	1,930	1,997	1,953
Morgan.....	10	1,915	1,995	1,966
Franklin.....	31	1,920	2,000	1,958
Center.....	13	1,907	1,995	1,955
Jackson.....	77	1,930	1,995	1,968
Aleppo.....	96	1,913	2,021	1,977
Springhill.....	115	1,952	2,034	1,988
Wayne.....	35	1,920	1,980	1,959
Whiteley.....	6	1,925	1,975	1,955
Jefferson.....	4	1,952	1,995	1,977

The distance of the Fifty-foot sand below the top of the Big Injun varies from 624 to 845 feet, as shown in the following table:

*Distances from top of Big Injun to Fifty-foot sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Richhill.....	31	690	845	768
Morris.....	11	710	815	755
Washington.....	6	715	805	766
Morgan.....	10	715	810	744
Center.....	13	715	772	755
Jackson.....	75	686	765	744
Aleppo.....	96	690	835	759
Franklin.....	30	690	800	735
Jefferson.....	3	730	765	744
Springhill.....	112	675	805	744
Giltmore.....	1			700
Wayne.....	33	624	725	688
Whiteley.....	4	704	735	711

In view of the fact that the interval between the Pittsburgh coal and the Big Injun sand decreases from south to north, it is interesting to note from the above table that the interval between the top of the Big Injun and the top of the Fifty-foot increases in the same direction. This southward thinning of the rocks below the Mauch Chunk about compensates for the above-mentioned northward thinning.

#### NINEVEH SAND.

The Nineveh sand occurs from 60 to 150 feet below the top of the Fifty-foot sand. It may be either white or red and ranges from 10 to 40 feet in thickness. It is the principal producing sand of western Greene County. In the Nineveh, Grays Fork, Bristoria, and Spring-

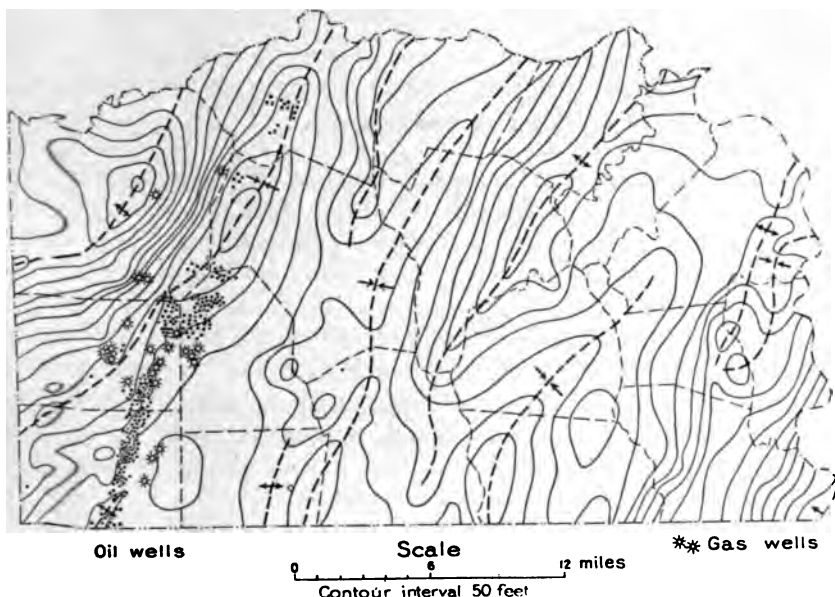


FIG. 3.—Structure map of oil and gas in Nineveh sand.

hill fields it is the principal oil sand and locally holds gas. One gas well is reported from it in Gilmore Township and it contains traces of gas elsewhere. Fig. 3 shows the present development of oil and gas in this sand.

The Nineveh sand has been erroneously called the "Gordon" and sometimes the "Gordon Stray" over the greater portion of the Nineveh district, as explained on pages 44-45 of this bulletin and in greater detail by F. G. Clapp in a paper on "The Nineveh and Gordon oil sands of western Greene County, Pa."<sup>a</sup> It takes its name from the village of Nineveh, in Morris Township, where it was first discovered to be productive. The sand is not reported in the eastern

<sup>a</sup> Bull. U. S. Geol. Survey No. 285, 1906, pp. 362-366.

part of the county. Red rock, commonly found directly below this sand, aids in identifying it. It lies from 1,997 to 2,121 feet below the Pittsburg coal, as shown in the following table. On account of mistaken identification in most of the original records, it was necessary to change the original names of the sands to the correct ones before calculating the averages.

*Distances from Pittsburg coal to Nineveh sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Morris.....	6	2,028	2,110	2,055
Richhill.....	32	1,997	2,054	2,036
Center.....	8	2,005	2,120	2,039
Jackson.....	66	2,024	2,065	2,044
Aleppo.....	101	2,018	2,098	2,056
Wayne.....	2	2,033	2,045	2,039
Giltmore.....	2	2,065	2,113	2,089
Springhill.....	116	2,033	2,121	2,053

#### GORDON STRAY SAND.

The Gordon Stray is a thin sand occurring in places a short distance above the true Gordon and at varying distances below the red rock associated with the Nineveh sand. The name has been often applied to the Nineveh, but such usage is obviously incorrect, as the Nineveh and Gordon sands are separated by red rock. The Gordon Stray seems to be the least definite of any of the commonly recognized sandstone beds; it may be either a split from the Gordon or a new sand. In the Delphene field and at a few isolated points elsewhere it produces gas. In the small Wright Run field, at several scattering points in the Bristoria field, and in one or two wells in the Board Tree field it produces oil.

#### GORDON SAND.

The Gordon sand lies at an average distance of 2,159 feet below the Pittsburg coal, or 200 feet below the top of the Fifty-foot sand, and is from 15 to 20 feet thick. In places it produces small amounts of gas and oil. The sand is usually recorded in wells which reach its depth.

As explained above, the term "Gordon" has been generally applied in western Greene County to the Nineveh sand, but such usage is incorrect. Also the Gordon has been erroneously called the "Fourth" sand by many drillers in the district. The Gordon is not generally productive in this area, but contains gas at one or two wells in western Aleppo Township and in southeastern Center Township, one or two in the midst of the Bristoria field, and a number of wells in the Richhill, Hoovers Run, and Waynesburg fields. Oil is found in

it in one or more wells in the Board Tree field, in one well on the southeastern edge of the Bristoria field, and in several wells on the western edge of the New Freeport field, in southern Aleppo Township.

Fig. 4 shows the location of the wells in which gas and oil have been found in the Gordon and Gordon Stray sands and their relation

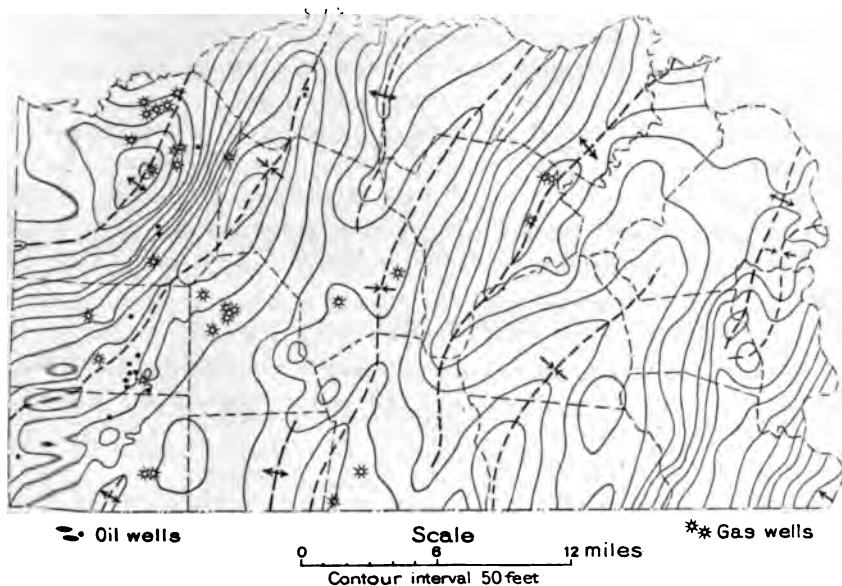


FIG. 4.—Structure map of oil and gas in Gordon and Gordon Stray sands.

to the approximate structure of the sand. In the following table the distances from the Pittsburg coal to the top of the Gordon sand are given:

*Distances from Pittsburg coal to Gordon sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Richhill.....	17	2,094	2,155	2,136
Morris.....	6	2,100	2,144	2,133
Washington.....	4	2,144	2,222	2,169
Morgan.....	4	2,145	2,184	2,163
Jefferson.....	1			2,205
Franklin.....	40	2,086	2,205	2,143
Center.....	8	2,083	2,170	2,127
Jackson.....	16	2,070	2,175	2,111
Aleppo.....	30	2,110	2,183	2,157
Springhill.....	3	2,130	2,205	2,166
Gilmore.....	8	2,177	2,189	2,181
Wayne.....	15	2,160	2,227	2,203
Whiteley.....	2	2,130	2,165	2,148

It will be seen by this table that so far as can be told from average intervals by townships there is no marked irregularity in the position of the Gordon sand with relation to the Pittsburg coal.

## FOURTH SAND.

Another sand which locally yields small amounts of gas or oil is the Fourth sand. It is reported at distances varying from 2,154 to 2,316 feet below the Pittsburgh coal and from 20 to 120 feet below the top of the Gordon sand. Its thickness ranges from 7 to 20 feet. The irregularity and occasional running together of the Gordon Stray, Gordon, and Fourth sands classes them as a group (the Gordon group) rather than as entirely distinct beds of sand.

In the Aleppo oil field the Fourth is the principal producing sand, and a number of wells have been drilled to it. Gas is produced from it at several wells on the borders of the New Freeport oil field, south of New Freeport and Deep Valley, and at some scattering wells in the Hoovers Run field and in Franklin, Jackson, and Richhill townships.

## FIFTH SAND.

The great gas producers in Greene County are the Fifth and Bayard sands. Practically all wells in the eastern part of the county,

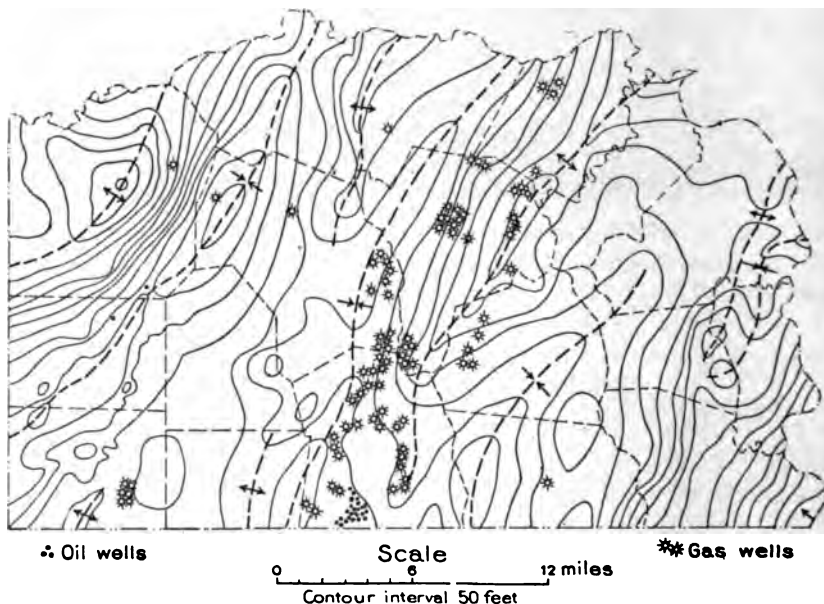


FIG. 5.—Structure map of oil and gas in Fifth sand.

except those in Dunkard, Greene, and Monongahela townships, are sunk to one or both of these beds. The Fifth sand lies from 2,225 to 2,365 feet below the Pittsburgh coal and ranges in thickness from 10 to 65 feet. It is known to some drillers as the McDonald sand, from the town of McDonald, Washington County. This sand yields some gas in the northern part of Morgan Township and in the Waynes-

burg field, and is the principal source on Pursley Creek and Hoovers Run. In the Hoovers Run field the Fifth sand occasionally goes by the name of Gas sand and in several instances has been called Gordon. In the Lantz field the Fifth sand yields oil from two pay streaks. Oil was found in this sand in one well at Waynesburg and also in the Aleppo field. Few of the wells in the western half of Greene County have gone deep enough to reach the Fifth sand, but it is known to yield gas in one well in Richhill Township and in two wells in northern Center Township.

The location of wells producing oil and gas from the Fifth sand is shown in fig. 5. Probably other wells in Franklin and Morgan townships found gas in the Fifth sand, but it was in small amount in comparison with the yield from the Bayard sand and the records do not mention the fact. A discussion of the curious relation of the oil and gas to the structure in Wayne Township will be found on page 82. The position of the Fifth sand with relation to the Pittsburg coal is shown in the following table:

*Distances from Pittsburg coal to Fifth sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Richhill.....	6	2,230	2,270	2,242
Morris.....	4	2,270	2,283	2,276
Washington.....	4	2,275	2,345	2,309
Morgan.....	5	2,292	2,365	2,316
Franklin.....	44	2,258	2,355	2,318
Center.....	8	2,225	2,335	2,260
Jackson.....	4	2,232	2,325	2,271
Aleppo.....	1			2,275
Springhill.....	1			2,294
Gilmore.....	2	2,335	2,352	2,344
Wayne.....	45	2,269	2,339	2,300
Whiteley.....	5	2,282	2,325	2,295

This table shows that in the eastern half of the county, at least, there is a marked parallelism between this sand and the coal, for in five townships the average distances vary only 23 feet. The following table shows the relation of the Fifth sand to the Big Injun in townships where it is reported:

*Distance from top of Big Injun to Fifth sand.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Morris.....	3	1,076	1,155	1,115
Morgan.....	5	1,075	1,150	1,109
Washington.....	4	1,075	1,123	1,101
Franklin.....	39	1,015	1,200	1,084
Center.....	9	1,035	1,112	1,075
Richhill.....	5	1,018	1,102	1,066
Jackson.....	3	1,035	1,086	1,059
Wayne.....	45	970	1,101	1,033
Whiteley.....	10	1,020	1,055	1,043

The figures in this table show a decrease in the interval from north to south. It is shown on page 34 that the interval from the Pittsburgh coal to the Big Injun sand decreases in the opposite direction, owing to an unconformity in the Mauch Chunk. A comparison of the intervals in six townships which form a broad area from the north to the southern boundary of the county is as follows:

*Average distances from top of Big Injun sand to Pittsburgh coal and Fifth sand.*

Township.	Pittsburgh.	Fifth.	Total.
	<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Morris.....	1,183	1,115	2,298
Washington.....	1,209	1,101	2,310
Center.....	1,208	1,075	2,283
Franklin.....	1,224	1,084	2,308
Wayne.....	1,268	1,033	2,301
Whiteley.....	1,257	1,043	2,300

The total of average distances for each township shows a rather striking parallelism between the Pittsburgh coal and the Fifth sand in this part of the county. It must not be inferred, however, that this regularity is at all constant in other sections. In western and northwestern Greene County the unconformity in the Mauch Chunk formation causes all intervals measured from the Pittsburgh coal to the lower sands to diminish rapidly toward the north and west.

#### BAYARD SAND.

In the eastern half of the county gas is found more frequently in paying quantities in the Bayard than in any other sand. In the western half of the county the Bayard horizon has been reached in only a score of wells. Of these, ten report the Bayard present and one found gas in it. A few have passed the horizon without recognizing it. Fig. 6 shows the large number of Bayard sand wells in the northeastern part of the county, on the Belle Vernon anticline.

In eastern Greene County the distance from the top of the Fifth sand to the top of the Bayard sand in 60 wells averages 120 feet, and the logs of the few wells which report the Bayard in the western part of the county record 130 feet between the same beds, the interval seems to be rather constant. The depth below the Pittsburgh coal varies from 2,388 to 2,464 feet, averaging about 2,433 feet for the six townships in the eastern half of the county, as shown in the following table:

*Distances from Pittsburg coal to Bayard sand in eastern Greene County.*

Township.	Number of wells.	Minimum.	Maximum.	Average.
		<i>Feet.</i>	<i>Feet.</i>	<i>Feet.</i>
Washington.....	3	2,446	2,457	2,455
Morgan.....	6	2,425	2,460	2,440
Franklin.....	44	2,388	2,464	2,435
Whiteley.....	4	2,405	2,430	2,414
Jefferson.....	3	2,400	2,415	2,405

The thickness of the Bayard sand seems to vary considerably. A few wells have passed completely through it and their logs give 3 to 12 feet as the thickness, except the Eaton well (83) in Jefferson

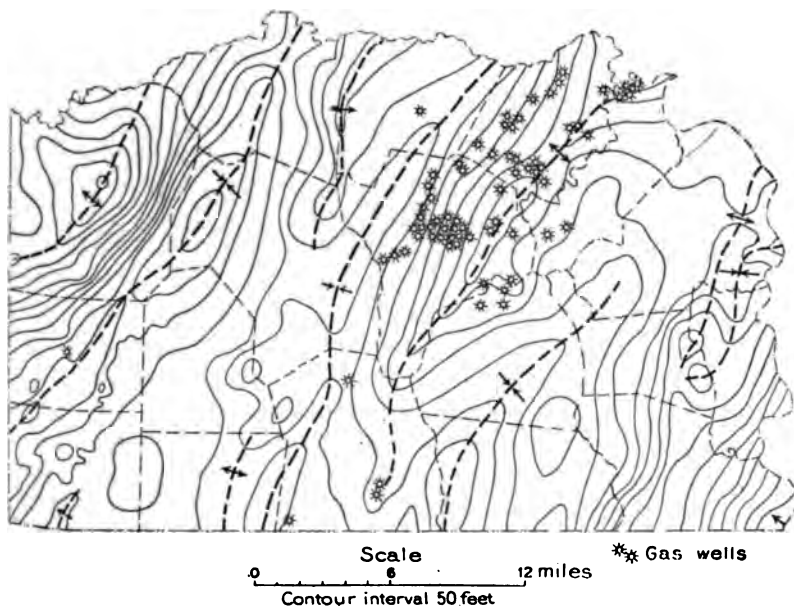


FIG. 6.—Structure map of gas in Bayard sand.

Township, in which 50 feet was recorded. In other wells which stopped when gas was struck and did not go to the bottom of the sand a thickness of 20 to 30 feet is noted. The Bayard is sometimes known as the Sixth sand, although this name has also been applied to the Elizabeth. The name Bayard was introduced in gas-sand nomenclature in February, 1895, when a successful well (167) was completed on the Thomas Bayard farm in Whiteley Township, Greene County.

#### ELIZABETH SAND.

This term is applied to a sand which carries some gas and is found at depths ranging from 100 to 175 feet below the top of the Bayard. Its thickness as recorded in three logs is not more than 7 feet; it rarely exceeds 10 feet, but in one well 12 feet of it are reported. Probably the number of holes sunk to this sand in Greene County is



less than 15. In the Warren Mankey well (101), near Nineveh, and in several wells farther south, the Elizabeth is reported as a red sand.

#### DEEPEST WELLS.

The deepest well ever drilled in Greene County is also the second deepest well in the United States. It was drilled in 1904 on the Benson heirs' farm (203), in western Aleppo Township, and reached a depth of 5,322 feet below the surface, or 4,722 feet below the Pittsburgh coal. This well recorded the Bayard sand 7 feet thick at a depth of 3,235 feet below the surface, and below this sand over 2,000 feet of rock are reported only as "shells and slate."

The second deepest well in Greene County was drilled on the Lewis Kuhn heirs' farm (204) in Wayne Township, and reached a depth of 3,780 feet from the surface, or 3,030 feet below the Pittsburgh coal. In this well a sand 15 feet thick was struck at a depth of 2,900 feet below the coal. This sand lies several hundred feet below the horizon of the Elizabeth sand and is provisionally correlated with the Speechley sand of Butler and Armstrong counties.

#### MISTAKEN IDENTIFICATION OF SANDS.<sup>a</sup>

In 1885, when the Devonian rocks were first penetrated by the drill in the oil field at Washington, Pa., the name Gordon was given to a sand about 250 feet below the top of the Gantz sand in a well on the Gordon farm. As drilling progressed southward the names used at Washington were naturally carried into Greene County. Thus it happened that when operations began in the Nineveh field the names Gordon and Fourth were applied to sands which were then believed to be equivalent to the beds called by those names at Washington. In eastern Greene County and in some areas in the western part of the county the names have been correctly used. This is true of a majority of the wells in Wayne Township, of a few in Center, Morris, and Springhill townships, and of most of the wells drilled by the Natural Gas Company of West Virginia in Richhill Township. Few records of other companies in Richhill Township are correctly correlated. Throughout the oil field extending southward from Nineveh beyond the West Virginia line, however, the name Gordon has been erroneously applied to a sand at the very top of the Catskill formation lying between the true Gordon and the Fifty-foot, and the name Fourth has generally been applied to the true Gordon sand. The name Gordon Stray has been used in that field to designate any bed occurring a short distance above what the drillers considered as Gordon.

In studying the stratigraphy of the Amity and Rogersville quadrangles a large number of well records were plotted on a uniform

<sup>a</sup>See also Clapp, F. G., The Nineveh and Gordon oil sands in western Greene County, Pa.: Bull. U. S. Geol. Survey No. 285, 1906, pp. 362-366.

scale and placed side by side in a manner similar to the arrangement on Pl. II (p. 22). A line of wells was taken, beginning at the original Gantz well, near the Gordon well in Washington, and extending southward to the Fonner field in Morris Township and thence to all parts of western Greene County. As can be seen by Pl. II, the Gordon sand was found to maintain its interval of 150 to 200 feet below the top of the Fifty-foot sand and to lie below the sand hitherto called Gordon in the Nineveh oil field.

In Richhill Township the names Nineveh Thirty-foot, Nineveh, or sometimes Thirty-foot, have been variously applied to the sand lying at the top of the Catskill formation. The name was taken from the village of Nineveh, near which the sand has produced oil. These identifications are correct, but for the sake of simplicity the word Nineveh alone will be used in this report. Hitherto the sand has been generally called Gordon throughout the oil field, but has sometimes been called Gordon Stray. The name Gordon sand will here be used in its correct sense as designating the sand more often called Fourth sand in this oil field.

By referring to Pl. II, the correctness of this tracing of the Nineveh sand from Washington into Greene County can be seen. It is not recorded in the Gantz well, but in the Baker well near Lone Pine, 8 miles southeast of Washington, it occurs 102 feet below the top of the Fifty-foot sand. In that region and frequently throughout eastern Washington County the name Gordon Stray is used for this sand. In the Baker well it attains an unusual thickness. The next section in line is that of the Fonner No. 2 well (205) in the Fonner field, Morris Township. In this well the Nineveh sand consists of 13 feet of red sand lying 48 feet below the top of the Fifty-foot, and a second red layer occurs 68 feet below it and a few feet above the Gordon sand. In the John Lewis well (206) in the same township the Nineveh sand was called Gordon, and from this point the sand can be traced almost continuously the whole length of the oil field, being known to the drillers either as Gordon or Gordon Stray.

In this connection the following statements should be made concerning the correlation of sands. It is recognized that the familiar names of gas and oil sands used by the drillers constitute a serviceable terminology, but it should be understood that the names indicate only approximate geologic position and not actual identity of sandstones. It can not be proven that the sands which are given the same name in different fields are continuous beds. In fact, the manner in which sandstone beds showing at the surface locally thin out and are replaced by shale or rocks of other character makes it probable that similar conditions exist in the deeper lying sandstones, so that these gas and oil reservoirs may be large lenses of sandstone rather than continuous beds.

**METHOD OF FINDING DEPTH OF SANDS.**

In using the contours represented on the structure map (Pl. ) should be remembered that few beds are exactly parallel, and allowance must be made for the increase and diminution of interval in various directions. The causes of variation are twofold—(1) increase or decrease in thickness of beds due to differences in sedimentation and (2) the marked changes in the Mauch Chunk formation resulting from an unconformity at its top. The thickness of Mauch Chunk decreases from southeast to northwest.

To get the depth of a sand below the surface at any point find the depth of the Pittsburgh coal by subtracting its elevation as shown on the structure contours from the elevation of the surface. Add to this the average depth of the sand below the coal as given in the table on page 31, or as given more accurately on a subsequent page in the discussion of that particular sand, and the result will be the approximate depth of the sand below the surface.

**DESCRIPTION OF THE OIL FIELDS.**

The oil fields of Greene County may be divided into two groups. The first group is in the southeast corner of the county and comprises the Dunkard Creek, Garrison, Whiteley Creek, Blacksburg, and Mount Morris fields. The western group is known as the Nineveh district, and includes a belt running from the northeast corner of Morris Township to the West Virginia line south of Deep Valley. This belt follows in a general way the axis of the Nineveh syncline and consists of the Fonner, Nineveh, Grays Fork, Wright I, Bristoria, Aleppo, New Freeport, and Board Tree fields. The Lantz field in Wayne Township stands alone.

**EASTERN GREENE COUNTY.****DUNKARD CREEK FIELD.**

Oil was first discovered in Greene County at a place once known as Bobtown, on Dunkard Creek, between  $2\frac{1}{2}$  and 3 miles above its mouth. Bobtown was the location of Maple's woolen mill a generation ago, but the mill has long been abandoned and demolished.

The first well was drilled on the Cephas Wiley farm (207) on the bank of Dunkard Creek, a few hundred feet above the covered bridge, which is 1 mile south of Wiley post-office. This well was begun in 1863 and finished in 1864, and the fact that it produced oil in considerable quantities started active drilling in this locality. Within a year drilling had progressed both up and down the stream.

and between 75 and 100 holes had been sunk to the Dunkard sand. Stevenson wrote of Bobtown in 1876 as being "in the immediate vicinity of a vast number of oil derricks." The oil in this field was found mostly in the Dunkard sand, at a depth of about 450 feet below the Pittsburg coal. Pay streaks carrying heavier oil were found occasionally in the Morgantown sandstone about 175 feet below the coal.

As the Pittsburg coal outcrops along the bank of the creek, the wells were not over 500 feet deep. Well drilling was a new business then and primitive methods were used. Wells were started by digging a hole with pick and shovel to bed rock and putting in a wooden conductor. The process of spudding seems not to have been known at that time. Drilling was done by means of a spring pole, a method which can be used only in "shallow wells." Many of the wells were drilled close to the outcrop of the coal, and all of them were in the flood plain of the creek or but slightly above it. Most of them produced more or less oil, although some wells but a few rods from producing wells proved to be dry holes. The wells yielded very abundantly for a time, but, having been drilled without casing, the soft shales were soon converted to mud by the flow of oil and water, and the holes caved, thus effectually stopping the flow. Had they been properly cased the field might have had a long life.

Several wells in the Dunkard Creek field were pumped for ten years or more. The oil was all barreled and hauled to the mouth of the creek, whence it was shipped by boat to Pittsburg. It is said that the original well netted its owner \$40,000. A well was drilled in 1865 on the B. Ross farm which produced a heavy oil from a sand about 175 feet below the Pittsburg coal. Twenty-four barrels of oil a day were pumped from this well for a number of months and sold for \$20 a barrel or retailed at 50 cents a gallon.

But few traces remain of this old field at present. In many cases the location of the well holes can be recognized only by a slight depression in the sod of the pasture or by the hollows in which the rig timbers stood. In fact, the Bobtown field is now but a matter of history.

This field is located at a point midway up the flank of the Fayette anticline, where for a short distance the rocks dip less than at any other place between Mount Morris and Monongahela River. Indeed, a slight fold crosses the creek, for the sandstone under the Pittsburg coal is seen dipping eastward at the covered bridge 1 mile south of Wiley. This fold is only a very minor feature in the structure, but may account for the accumulation of oil at this point.

In his report on Greene County, published in 1876, Stevens gives the following notes on this field:

The productive oil territory begins about 2 miles above the mouth of Dunkard extends along the line of the creek for barely 2 miles, including the Elliott, Maple, rison, Bailey, and Ross farms. For the most part the wells were bored on the bottom and the curbs are only a few feet below the Pittsburg coal.

Elliott farm: Three wells were bored here, all of which were productive. One of the Bobtail, flowed for nearly three weeks, giving an average of 80 barrels per day the flow gradually diminished and at last ceased. Its total yield is not far from barrels. Only 1 well is now in operation, and that gives to the pump 1 or 2 barrels day. The oil comes from the lower horizon, about 400 feet below the Pittsburg coal each of the wells some oil was found at the upper horizon, but the quantity was very

Maple farm: Between this and the Elliott farm wells were bored at several localities but they proved either insignificant or utterly barren. A great number of borings made on the Maple farm, and it was the main center of the oil excitement. The territory was extensive, but in the absence of all records I am unable to estimate it even approximately. The Wiley well No. 1 has produced more than 5,000 barrels and still yields a little each day. The Allegheny well at first flowed at the rate of 125 barrels per day but the flow gradually decreased until the well ran dry. The Lone Star still yields a quantity—2 or 3 barrels a week. It flows on provocation, pumping being needed to stir up the gas. The oil was obtained on this farm at two horizons—175 and 440 feet below the Pittsburg coal. Salt water was found at various depths, but no tests were made to determine its quality.

Garrison farm: This adjoins the Maple farm northward and includes Deep Run where the Butler well was bored. This well, like the Lone Star, flowed upon provocation and yielded in all about 1,500 barrels. The flow was stopped by caving of the walls. The oil was obtained here at the upper horizon, and very little water, either salt or fresh encountered in the boring.

Bailey farm: This adjoins the Maple farm along the creek. Only three wells were bored here. No. 1 found a good supply at the upper horizon and yielded at first from 30 barrels daily. It now gives only 1 or 2. No. 2 has proved almost unproductive. No. 3 is known as the "Drywell," as no oil and very little water have been found. Good brine, said to be 10° strong, was obtained from Nos. 1 and 2.

Ross farm: This is next above the Bailey farm on the creek. Of the 15 wells put down here, 12 are on the "bottom" beginning at about 10 feet below the Pittsburg coal. Oil was obtained in large quantities at both horizons. The first well, known as Ross No. 1, flowed 200 barrels during the first day, and then suddenly stopped, as the walls had fallen. A similar mishap befell several other wells which opened in an equally promising manner. Two are now in operation, which yield 7 to 8 barrels per week from the lower horizon.

Borings have been made at several localities along the creek above the Ross farm but they have proved uniformly unsuccessful. As no oil has been obtained at any locality below the Elliott farm, it is easy to define the limits, north and south, of the productive territory.

Oil from the upper horizon, 170 to 180 feet below the Pittsburg coal, is uniformly heavy usually about 32°. The only exception is the Bailey well No. 1, in which the gravity is 34°. The lower horizon, in the Mahoning sandstone, yields a much lighter oil of 40° to 42° gravity. This oil is much valued in the raw condition for cleaning wool, and when reduced by exposure or steaming to about 34° it becomes a lubricating oil of excellent quality. Some of the inhabitants crude oil is used for illuminating purposes. Experience leads me to assert that it is not well adapted to that use.

<sup>a</sup> Second Geol. Survey Pennsylvania, Rept. K, 1876, pp. 100-102.



1. OIL WELLS ON DONLEY FARM NORTHERN EDGE OF MOUNT MORRIS VILLAGE



2. GROUP OF OIL WELLS IN GARRISON FIELD.

Original Tanner well in foreground.



## GARRISON FIELD.

After a lapse of several years drilling was attempted again a few miles farther north. In June, 1876, Abner P. Tanner, of Pittsburg, put down a well (208) 2 miles south of Willow Tree, on the Garrison farm, at the point where the highway crosses the run. The result was a 50-barrel well in the Dunkard sand. So great was the excitement that people came from far and near to see it, and it is said that on an occasional Sunday 500 people visited the spot. For twenty-eight years, or until 1904, this well was pumped steadily, the yield falling off gradually until it amounted to so little that the casing was pulled and the well abandoned. Although this well was successful, so far as can be learned no further drilling was done until 1889, when several holes were put down. Of recent years drilling has been going on almost constantly in this vicinity, and there are now about 70 wells in the field, 47 holes having been drilled on the Dan Garrison farm. This field is also known as the Tanner field, in honor of the man who sunk the first well. Pl. III, A, shows a group of wells in the Garrison field, with the original Tanner well in the foreground.

The total production at present for this field is about 25 to 30 barrels a day. Out of 47 wells on the Garrison farm, 16 are producing oil at present. Many of the others yielded oil when first drilled, but have been pumped dry. Besides these, 2 wells on the Alfred Harrison farm, 1 on the Sycks farm, 4 on the Cumston farm, and 1 on the Long farm are producing small amounts of oil. For years the oil from this field was hauled to Monongahela River in barrels, but it is now transported by pipe line. Structurally the Garrison oil field is located on a small, gently sloping bench at the south end of the Lambert syncline. It is at the top of the steep grade in the vicinity of Davistown, where the dip changes from more than 100 feet to less than 50 feet to the mile.

## WHITELEY CREEK FIELD.

The Whiteley Creek field lies on Whiteley Creek, in the southeast corner of Greene County, about halfway between Whiteley and Mapletown. A well was drilled at Vance's mill (212), at some time previous to 1876, in regard to which Stevenson<sup>a</sup> says:

This well was bored by Mr. G. Vance, who obtained oil at 120, 368, and 395 feet below the Pittsburg coal. When the lowest horizon was reached, oil of light specific gravity, about 40°, flowed at the rate of 2 barrels per diem; but soon, as in so many other instances in this region, the walls caved and the flow ceased. An attempt to clean it out was made, but was unsuccessful, resulting in the loss of two sets of tools which, with 100 feet of rope,

<sup>a</sup> Second Geol. Survey Pennsylvania, Rept. K, 1876, p. 122.



still remain in the well. Notwithstanding all these obstructions, there is an energetic flow of gas, and oil is obtained whenever pumping is resorted to. Oil from the top horizon was heavy and that from the bottom was light.

In later years 5 or 6 other wells have been drilled at this old mill. The tools stuck in a second well and 1 was dry, but 2 are still being pumped.

In 1885, more than twenty years after the Bobtown development E. M. Hukill, of Pittsburg, acquired leases covering about 60,000 acres, extending from Rices Landing, on Monongahela River, southward thru Greene County and into Monongalia County, W. Va., as far as Pawpaw Creek. Drilling along this line resulted in the discovery of oil at Willow Tree, on the farm of Stephen Garard, in 1886. The first well on the farm was begun 50 feet below the Waynesburg coal and was drilled to a depth of 2,500 feet without getting oil. A second well (209) was drilled on the same farm, 1,000 feet east of No. 1, and oil was found in paying quantities in the Dunkard sand, which is 450 to 480 feet below the Pittsburg coal in this locality. A small field was developed in the immediate vicinity, a number of other wells being sunk on the Gregg farm (68). Most of these were successful, some yielding as high as 100 barrels of oil a day at the start.

About 1895 J. L. Garard began drilling for oil at Willow Tree and has sunk over 40 wells, most of them within half a mile of the crossroads. Up to the close of 1905 about 50 wells had been drilled in the Whiteley Creek field. About one-third of these were dry; the rest produced oil in varying amounts. Some of the wells which yielded oil at first have gone dry, but nearly half of the wells in the field are being pumped and yield a total of about 80 barrels of oil a day. The producing wells are on the following farms: J. L. Garard (209), 3; J. C. Gregg (68), 10; W. L. Longanecker (210), 8; N. H. Minor (211), 1.

The Whiteley Creek field is located at the south end of the Lambert syncline. Near the oil field is a shallow local basin, with its center about three-quarters of a mile east of Willow Tree. From this point the rocks rise gently in all directions, the most marked rise being to the west. The wells of the Whiteley Creek field are located on this westward rise, the altitudes of the rock strata varying from 30 to 60 feet above their position at the center of the basin. The log of the original Gregg well is given herewith.

*Record of Gregg well (68), one-half mile south of Willow Tree.*

	Thick- ness.	Depth.
	Feet.	Feet.
Limestone.....	60	60
Slate and slaty sandstone.....	30	90
Coal, Mapletown.....	5	95
Limestone with slaty partings.....	60	155
Shale, black, and cannel coal.....	20	175
Sandstone, coarse.....	50	225
Coal, Pittsburg.....	9	234
Slate.....	30	264
Limestone shells.....	20	284
Slate.....	40	324
Sandstone, coarse (water).....	20	344
Slate.....	50	394
Sandstone ("First Dunkard").....	80	474
Slate, white.....	70	544
Red rock.....	70	614
Slate, white.....	15	629
Sandstone.....	15	644
Slate, white.....	40	684
Sand (Dunkard).....	30	714
Slate.....	10	724
Sandstone, white (Mahoning).....	66	790
Sandstone, black.....	16	806
Slate and shells.....	86	892
Sandstone, white.....	32	924
Slate and shells.....	120	1,044
Slate.....	20	1,064
Sandstone, black (oil show).....	17	1,081
Coal.....	8	1,089
Sandstone, white (salt water at 1,109 feet).....	50	1,139
Slate.....	35	1,174
Sandstone, white (salt water at 1,209 feet).....	90	1,264
Red rock (Mauch Chunk).....	145	1,409
Sandstone, white (gas and salt water at 1,587 and 1,629 feet) (Big Injun).....	320	1,729
Slate and shells.....	15	1,744
Slate.....	30	1,774
Sandstone, red (oil show).....	39	1,813
Slate and sand shells.....	140	1,953
Sandstone, gray.....	36	1,989
Slate and shells.....	95	2,084
Sandstone, brown.....	30	2,114
Slate and pebbles.....	15	2,129
Sandstone, gray, supposed to be the Gantz sand.....	25	2,154

#### BLACKSHIRE FIELD.

The Blackshire is a pool of very limited area situated on Whiteley Creek at the point where it is crossed by the direct road between Mapletown and Sigsbee, Monongahela Township. The original Blackshire well (213), drilled about 1865, gave 100 barrels or more a day at the start and was the incentive for the sinking of a number of other wells surrounding it. The production of the original well rapidly declined and soon ceased. Oil was found in the Big Injun sand at depths of 1,250 to 1,350 feet below the Pittsburg coal. A second well (214), located a few rods below the bridge, is said to have yielded to the pump at first 80 barrels a day. A well drilled about 1865 on the Stilwell farm (215), a quarter of a mile below the bridge, is said to have gushed over the derrick for three or four hours, but the oil softened the shale and the well caved about a day after it was finished. A number of wells were drilled to the Dunkard sand along Whiteley Creek east of Sigsbee and up the creek toward Mapletown, but none produced either oil or gas. The pool is located on the outer portion of the western slope of the Fayette anticline. The dips are

very gentle, though the rocks show a perceptible pitch to the north west. In the immediate vicinity of the wells there appears to be local flattening, which interrupts the general northwestward dip and may account for the occurrence of oil at this point.

#### MOUNT MORRIS FIELD.

The oil field which terminates in Dunkard Township, between Glade Run and Bowen Fork, three-quarters of a mile west of Dav town, is the northern extension of the great Mannington-Mount Morris pool. This pool is marked by a continuous line of producing wells from a point about 6 miles southwest of Mannington, W. V. to Dunkard Creek, a distance of 35 miles. It lies at the foot of the western limb of the Fayette anticline.

The development of the Mount Morris field dates from October 1886, when E. M. Hukill got a 20-barrel well (106) in the Big Injun sand. This well was located on the D. L. Donley farm, on Mor Run, half a mile southeast of Mount Morris, in Perry Township. In 1887 the tools were put in again and the well was drilled to the deep sands, without, however, increasing the production. In November 1887, the lower part of the hole was plugged and a torpedo exploded in the Big Injun with satisfactory results. A well (216) drilled in 1888 on the same farm one-eighth of a mile east of the original well produced 350 barrels of oil daily at first. Practically all the wells drilled on the eastern side of the village were successful and have been pumped for many years. A hole (217) sunk on the Asa Lemley farm in 1888 was thought at the time to be good for 5 barrels of oil a day but this was too small a production to bother with, and after drilling thru the Big Injun the well was abandoned. The map shows a deep hole (218) on the Williams farm at the mouth of Calvin Run and another (219) one-half mile to the southeast, on the place of J. Donley. These holes seem to be in productive territory, but were never completed because the tools were lost.

The Dunkard Oil and Gas Company was organized in 1892 and developed the field northward into Dunkard Township. The pool is about 1,000 feet wide and very rich. Wells drilled east of the belt and higher up on the flank of the Fayette anticline yield gas.

Oil in this pool is found in the Big Injun sand. The records of wells in Dunkard Township show that the first pay streak is reached at 1,367 to 1,392 feet below the Pittsburg coal, or about 100 feet below the top of the sand. The average distance between the coal and the oil horizon in the 13 wells is 1,377 feet. What is known as the "oil pay" or "pay streak" is a soft, porous part of the formation filled with oil and gas. In the early days the rock pressure in the pay streak was 350 pounds to the square inch, but it has fallen off

about 80 or 100 pounds. In the oil belt the "pay" has a maximum thickness of 8 feet and a minimum of 6 inches. It thins out as the western boundary is approached and thickens toward the southeast, having at several of the big gas wells east of the oil belt a thickness of 35 feet. Some wells in the Big Injun sand have two pay streaks, which are separated by 3 to 6 feet of very hard sandstone. There is no water in the upper part of the Big Injun, but salt water is found from 14 to 20 feet below the oil. At no place in this field, however, has the water been of sufficient quantity to interfere with drilling to the deep sands, nor does there seem to be a greater amount of water in the syncline within a mile west of the oil belt. Nearly all wells in this field have been drilled to the salt-water horizon and pump some brine. There is an impression prevalent that as the production of oil decreases the flow of salt water increases; this, however, is easily explained by the fact that when a well is yielding 50 to 100 barrels of oil a day, a small amount of water is scarcely noticed, but when the oil falls off to 2 barrels a day, the same amount of water becomes more evident.

The Mount Morris oil belt, as mentioned above, is at the base of the western limb of the Fayette anticline. It will be noticed on the map (Pl. I) that the line of producing wells follows closely the 550-foot contour on the Pittsburg coal. East of this belt the dip of the strata for a short distance is about 125 feet to the mile, while west of it the dip is about 50 feet to the mile. This change in the dip may be the cause of the accumulation of oil along this line, and the structure suggests that a continuation of the pool may be found by drilling on Meadow Run at the road forks  $1\frac{1}{4}$  miles above Davistown.

Within half a mile of the hotel at Mount Morris about 50 wells have been drilled and more than half of them are being pumped at the present time. Northward from the village the principal oil wells are on the Donley, Patterson, McClure, Stoneking, Pride, and Litman farms. Pl. III, B (p. 48), shows a group of wells on the Donley farm.

A pipe-line system controlled by the Standard Oil Company transports the product of the field to the seaboard. Pump stations are located at Mount Morris, Dolls Run, Jakes Run, Fairview, and Mannington. The oil is pumped from these stations to a central station on Monongahela River 2 miles above Morgantown, W. Va.

The character and succession of the rocks in this field are shown in the following log, which is the record of one of the early wells drilled near Mount Morris.<sup>a</sup> This well is south of the village, a few rods over the State line in West Virginia.

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<sup>a</sup> Bull. Geol. Soc. America, vol. 3, 1901, p. 189.

*Record of Core well No. 2, Monongalia County, W. Va.*

	Thick- ness.	I
Conductor.....	<i>Feet.</i> 21	
Slate.....	104	
Sandstone (Waynesburg).....	45	
Coal (Waynesburg).....	10	
Limestone and shale.....	120	
Sandstone.....	25	
Limestone (Benwood or "Great" limestone).....	85	
Slate, black.....	10	
Coal (Sewickley?).....	10	
Limestone.....	85	
Coal (Pittsburg).....	10	
Slate.....	70	
Sandstone.....	55	
Shale, red.....	35	
Sandstone.....	15	
Shale, red.....	10	
Shale, blue.....	25	
Sandstone (Morgantown).....	55	
Slate, blue.....	40	
Slate, blue and red.....	20	
Limestone and hard beds.....	80	
Slate, red.....	5	
Sandstone.....	25	
Slate, dark.....	60	
Sandstone (Mahoning).....	30	
Slate, light gray.....	60	
Sandstone (Freeport).....	80	
Slate, dark.....	25	
Limestone.....	40	
Slate, dark.....	40	
Sandstone, hard.....	5	
Slate.....	60	
Sand, Salt.....	150	
Slate.....	10	
Limestone (?).....	20	(Pottsville)
Slate.....	10	
Sand, dark pebbly.....	20	
Sandstone, light colored.....	95	
Limestone, hard.....	22	
Shale, red.....	13	
Slate, dark.....	45	(Mauch Chunk)
Shale, red.....	3	
Limestone (Greenbrier).....	56	
Sand, Big Injun (Pocono) (oil).....	101	

## WESTERN GREENE COUNTY.

## FONNER FIELD.

The Fonner field is in the northeast corner of Morris Township the western flank of the Amity anticline, and extends northward Washington County. The wells in Greene County are on the Fon Brooks, Bristol, and Shoup farms.

Oil was discovered in this field in March, 1897, in a well (drilled on the farm of William Fonner. This first well produced 1,800 barrels a day for a short time and then declined. A number of wells were drilled in 1898-99 to the Gantz sand, which yields oil. In several cases gas was found just above the oil. The average depth of the oil-bearing sand below the Pittsburg coal shown by the records of 7 wells, is 1,943 feet. The Pittsburg (reported in the Fonner No. 5 (98) to be 7 feet thick) is 400 to feet above sea level in this field, and the elevations of the well may vary from 1,180 to 1,500 feet above tide. This gives a variation in the depth of the Gantz sand below the surface from 2,680 nearly 3,000 feet, depending on the elevation of the well.

A number of dry holes were drilled in territory immediately adjoining productive wells. There were 10 producing wells in the Fonner field in February, 1903, with a total daily yield of about 50 barrels. The oil is pumped into small tanks and transported by the South West Pennsylvania Pipe Lines to storage tanks at Meadowlands, Washington County.

A typical well record from this field is given below:

*Record of Fonner well No. 5 (98), Morris Township.*

	Thick- ness.	Depth.
	Feet.	Feet.
Slate and lime.....		510
Coal (Waynesburg A).....	5	515
Slate and lime.....	45	560
Coal (Waynesburg).....	5	565
Slate and lime.....	330	895
Coal (Pittsburg).....	7	902
Lime and slate.....		1,160
Cave, very bad.....	120	1,280
Limestone, black.....	20	1,300
Slate and sand.....	65	1,365
Sand and lime.....	449	1,814
Sand, Gas (top).....		1,814
Sand, Salt (top).....		1,914
Lime, Big.....	21	2,100
Sand, Big Injun.....	225	2,325
Slate and shells.....	375	2,700
Sand, Thirty-foot.....	108	2,808
Sand, Gantz (top) (gas at 2,830 feet; oil at 2,832 feet).....		2,808
Sand, blue.....	35	2,869

This well was abandoned in 1899.

#### NINEVEH FIELD.

Between 15 and 20 oil wells, situated within  $1\frac{1}{2}$  miles of Nineveh, Morris Township, have constituted the Nineveh field. The oil has come for the most part from the Nineveh sand, but seems in two or three instances to be derived from the Gordon Stray.

Oil was struck in quantity in this field July 26, 1888, in the John H. Smith No. 3 well (104) of the Nineveh Petroleum Company, situated 0.8 mile northwest of Nineveh. This well, according to Carll<sup>a</sup>—

when drilled 20 feet in the sand, struck oil and flowed 3,940 barrels during the first thirty days. In attempting to clean it out and drill through the sand, a string of tools was dropped. A long fishing job ensued, which was further complicated by the burning of the rig November 15, 1888, and the loss of other tools. Finally, after there had been anchored in the hole one full set of  $4\frac{1}{4}$ -inch tools, one full set of  $5\frac{1}{4}$ -inch tools, and one  $5\frac{1}{2}$ -inch bit, in all about 130 feet of iron, fishing was abandoned. A string of  $4\frac{1}{4}$ -inch casing was then let down to the obstruction, and through this a new hole was drilled by the side of the fastened tools, and without disturbing them, down to and through the sand. Oil was struck the second time March 16, 1889, and the well again started to flow at the rate of 310 barrels per day without a torpedo.

<sup>a</sup>Carll, John F., Seventh report of the oil and gas fields of western Pennsylvania: Second Geol. Survey Pennsylvania, Rept. I 5, 1890, p. 310.

The eastern portion of this field lies in the bottom of the Nineveh syncline, but the greater part of the wells are on the western side of the axis, less than 50 feet above the bottom of the trough.

#### GRAYS FORK FIELD.

Southwest from the Nineveh field, along the strike for nearly miles, a number of holes were drilled in a fruitless endeavor to continue the productive area in that direction. In the vicinity of Grays Fork, at the south end of this line of dry holes, a few wells nearly a mile west of the synclinal axis have produced oil from the Nineveh sand.

#### BRISTORIA FIELD.

Southwest of Grays Fork is another gap of 3 miles in which only a few dry holes have been drilled. Between Bristoria and Delpher and in the vicinity of Higbee is the Bristoria field, covering an area of about 6 square miles, in which a large quantity of oil has been produced since 1896. This field lies in portions of Jackson, Aleppo, Richhill, and Center townships, and occupies the valley and interstream areas of Job Creek and Long Run. It is almost entirely on the east side of the syncline, though a few productive wells lie west of the axis. Most of the oil comes from the Nineveh sand, although on or more wells on the southeastern border of the field obtain it from the Gordon. Scattered throughout the field are a few gas wells, and to the southeast, on the anticlinal slope in the vicinity of Delpher, lies a small gas field. Most of the gas is produced from the Nineveh, Gordon Stray, and Gordon sands, and in one or two wells from the Fourth sand.

#### NEW FREEPORT FIELD.

In Aleppo Township, south of the Bristoria field, there is an area about  $1\frac{1}{2}$  miles long, parallel with the syncline, in which little oil is found. Beyond this area the productive belt is continued southward thru Aleppo and Springhill townships to the State line south of Deep Valley. This field also has been operated since 1896. As in other fields in the belt, the Nineveh is the productive sand, although a few wells obtain oil in the Gordon. In the New Freeport field the Salt, Gordon, and Fourth sands produce some gas, and on the eastern side of the oil field, from New Freeport southward, lies a gas field in which the Fifth sand is the valuable bed.

#### BOARD TREE FIELD.

The Board Tree field lies about 2 miles west of the New Freeport field and is separated from it by a minor anticlinal fold. Most of the oil in this field comes from the Big Injun sand. The Nineveh

and Gordon sands have also produced small quantities of oil in this vicinity. High up on the anticlinal slope, southeast of this field, several wells produce gas from the Big Injun and Salt sands.

#### ALEPPO FIELD.

There is a small group of wells lying on the west side of the Nineveh syncline in which oil is obtained mainly from the Gordon and Fourth sands. Some gas is found in the Fifth and Nineveh sands, along the upper border of this field, and one well reports oil in the Fifth.

#### WRIGHT RUN FIELD.

Three wells on Wright Run,  $1\frac{1}{4}$  miles northeast of Bristoria, constituted the Wright Run field, when last visited in 1904. The oil in these wells is believed to occur in the Gordon Stray or upper part of the Gordon sand. They are light wells, and one of them produces gas. On account of its position, halfway up the steep anticlinal slope, the field probably does not contain much oil.

#### LANTZ FIELD.

The Lantz pool is the north end of a large field extending over the State line into West Virginia. It lies in Wayne Township, near the junction of Hoovers Run and Dunkard Creek, just east of the village of Shamrock. Operations in this field date from 1899, when the William Lantz No. 1 well (231) was drilled. Most of the oil is produced from the Fifth sand, which also yields considerable gas in Wayne Township. Some drillers have erroneously called this sand the Gordon, and some name it the Gas sand.

The position of this oil pool, higher up the limb of the syncline than the gas field on the west, is a curious feature that is discussed on page 82.

#### MISCELLANEOUS WELLS.

Besides the wells in the above-described fields a number of others in the eastern half of the county have obtained oil in small amounts. Two wells drilled in 1893 on the Lantz farm at the mouth of Woods Run, Greene Township, have an interesting history. According to a letter from C. E. Lantz, written in December, 1905, the well (220) in the road corner tapped the first pay streak in the Big Injun sand and filled up 300 feet with oil. The tools were lost in the hole and a set of fishing tools lost on top of them. Some oil was removed by the sand pump. A second well (221) was drilled on the same farm, on the bank of Whiteley Creek, and went to the Bayard sand, getting only a small pocket of gas in the Big Injun. The third well (222) was drilled on Woods Run, a few rods above the first, and is said



to have filled up about 100 feet with oil when the Big Injun pay was tapped. Because of some irregularity in the business transaction these wells were never pumped. If oil appeared in these wells, described, it may be considered good evidence that the continuation of the Mount Morris pool should be sought in this direction, for line of equal elevation on the producing sand in that pool passes close to the mouth of Woods Run.

A small showing of oil was found in the Big Injun sand in a well (223) on the A. Rice farm, three-fourths of a mile west of Whitesburg. The L. L. Thomas well (164), at Blacksville, produced some oil from the Fifth sand.

A well (224) near the pottery at Greensboro was started about 1 foot below the outcrop of the Pittsburg coal and 30 feet above Monongahela River. The Mahoning sandstone, or Dunkard sand was encountered at 300 feet, and it is said to have yielded about 1 barrel of oil a day. The Big Injun sand yielded some gas and showed oil.

Oil wells on the Brown and Shriver farms on Dunkard Creek, the mouth of Blacks Run, are in West Virginia. The Molesey well (225),  $1\frac{1}{2}$  miles east of Jefferson, obtained some oil from the Dunkard sand. This well was sunk to the Elizabeth sand, which proved to be a good gas producer. The Ellen Ross well (226) on Ruff Creek, five-eighths of a mile above Ruff Creek post-office, obtained about 1 barrel of oil daily from the Gantz sand. The flow of gas in this well was weak. A well (56) on the land of E. M. Sayers, at Waynesburg, got a trace of oil in the Fifth sand. Oil was found also in small quantities in the J. Marshall well (227), near Brock. None of these wells yielded enough to pay for pumping. The Headley well (228) on Little Shannon Run, is said to have made a good showing of oil, but when the well was shot the oil was drowned by salt water. The record does not state in what sand this oil was found. On the Joseph Morris farm, 3 miles northwest of Blacksville, W. Va., a well (229) got oil in the Gordon sand and was pumped several years. In 1904 it was drilled down to the Fifth sand and is now producing from the lower horizon.

The record of the H. M. Spragg well (162), which was drilled by the South Penn Oil Company, one-half mile south of Spraggs, Waynes Township, in 1895, and showed a trace of oil in the Big Injun sand is given on the next page.

*Record of H. M. Spragg well (162), Roberts Run.*

	Thick- ness.	Depth.
	<i>Feet.</i>	<i>Feet.</i>
Coal, Pittsburg (top).....		815
Sand, Dunkard.....	70	1,200
Sand, Gas.....	30	1,525
Sand, Salt.....	110	1,690
Sand, Big Injun (gas at 2,116 feet; oil at 2,130 feet).....	254	2,150
Sand, Thirty-foot.....	30	2,425
Sand, Gantz (gas at 2,550 feet).....	27	2,573
Sand, Fifty-foot.....	45	2,630
Sand, Gordon.....	14	2,853
Sand, Fourth.....	7	2,920
Sand, Fifth.....	15	2,946
Total depth, steel-line measurement.....		3,215

### DESCRIPTION OF THE GAS FIELDS.

In earlier reports on Greene County the distribution of gas has been discussed by fields, but now that intervening territory has been drilled and found productive, some of the small fields have become merged. In such cases no well-defined lines can now be drawn between the separate groups of wells formerly described as fields, and therefore it seems best to discuss the distribution of gas by townships. They will be taken up in geographic order from Monongahela River westward.

#### CUMBERLAND TOWNSHIP.

So far as has been learned only 6 deep holes have been drilled in Cumberland Township, and all were unproductive. Three of these are on Muddy Creek—the Biddle well (31), one-half mile below Carmichaels, 2,432 feet deep; the Rea well (232), 1 mile above Carmichaels; and the Rich well (233) at Khedive, said to be 3,100 feet deep. D. C. Stevenson drilled a well (234) at Ceylon in 1864 and got a showing of oil. This well was 700 feet deep, the curb being 36 feet below the Waynesburg coal. The following paragraph relative to this hole is from the report of the Second Survey:<sup>a</sup>

At 288 feet the Pittsburg coal was reached, giving the interval between the coals as 324 feet, which is much less than at any locality south from this where direct measurements have been made. Oil was obtained at 460 and between 600 and 700 feet. These answer to the first and second horizons of the Dunkard wells. Brine was found in close proximity to the oil. The lower stream was strong and yielded excellent salt, but no thorough test was ever made to determine the strength of the brine, nor has anyone attempted to utilize it by the manufacture of salt.

Mr. Stevenson writes under date of January 25, 1906:

The well was completed in 1864. At that time we had no casing, and the water was hard to shut off. The hole was drilled 250 feet with a 4½-inch bit and 450 feet with a 4-inch bit. Salt water was struck and flooded the well within 6 feet of the surface. It could not be exhausted with the appliances we had. All the oil we got was by means of the sand pump.

<sup>a</sup> Stevenson, J. J., Second Geol. Survey Pennsylvania, Rept. K, 1876, p. 127.

A well was drilled at the mouth of Little Whiteley Creek many years ago, but its exact location is not known. The record of one well in this township is available, and is given below:<sup>a</sup>

*Record of Biddle well (31), Cumberland Township.*

	Thick- ness.	De-
	Feet.	F
Conductor.....	7 1/2	
Limestone and layers of slate.....	48 1/2	
Limestone, soft.....	147	
Limestone, hard.....	62	
Slate and shell.....	35	
Sandstone.....	30	
Slate.....	5	
Coal, Pittsburg.....	9	
Slate and shells.....	176	
Sandstone (salt water).....	50	
Slate and shells.....	62	
Sandstone.....	23	
Slate.....	40	
Red rock.....	5	
Slate.....	95	
Sandstone.....	35	
Slate.....	35	
Sandstone, "Dunkard Third sand".....	40	
Slate.....	4	
Sandstone.....	11	
Slate.....	35	
Slate and shells.....	80	1
Sandstone, dark gray (gas at 1,040 feet; salt water at 1,050 and 1,180 feet).....	165	1,
Slate and shells (1,167 feet of 7 1/2-inch casing).....	35	1,
Sandstone (fresh water at 1,240 and 1,250 feet).....	190	1,
Slate.....	10	1,
Limestone.....	75	1,
Red rock, red shale.....	25	1,
Limestone.....	60	1,
Sandstone, "Big Injun" (salt water at 1,600 feet; some gas and oil; 1,613 feet of 5 1/2-inch casing).....	240	1,
Slate and shells.....	40	1,
Sandstone, hard.....	35	1
Slate.....	80	1
Sandstone.....	220	2
Slate and hard sand shells.....	10	2
Slate.....	210	2
Red rock to bottom.....	12	2

MONONGAHELA TOWNSHIP.

Aside from the oil wells and dry holes in the Dunkard Creek and Blackshire fields, described on pages 46-48, 51-52, but few deep holes have been drilled in this township. Most of these are on the banks of Monongahela River. Wells above and below the mouth of Whiteley Creek on the Gray (235) and Keener-Durr (236) farms found some gas in the Gantz sand. Gas has also been obtained on the Silas Ross farm (237) at the mouth of Dunkard Creek, and in a single well on Whiteley Creek, 1 mile west of Mapletown. Four wells were drilled in the northern outskirts of Greensboro in 1904, and three of them are producing gas from the Big Injun sand. These are the Mary Reed (238), John Steele (239), and B. F. Gabler (240) wells. A well (88) on the Williams farm, one-half mile southwest of Greensboro, also drilled in 1904, was dry, getting only a show of gas, tho it started at the Pittsburg coal and went down 2,120 feet.

The record of a dry hole drilled by the Greensboro Gas Company on the land of Dr. Ben Birch, one-half mile north of Greensboro, is exceptionally good and is given on the next page.

<sup>a</sup> Second Geol. Survey Pennsylvania, Rept. 15, 1890, p. 315.

*Record of Dr. Ben Birch well (86) near Greensboro.*

	Thick- ness.	Depth.
	<i>Feet.</i>	<i>Feet.</i>
avel.....	37	37
.....	40	77
.....	10	87
.....	23	110
.....	15	125
.....	38	163
.....	10	173
.....	30	203
.....	25	228
.....	28	256
.....	30	286
.....	3	289
.....	11	300
nd water.....	5	305
.....	8	313
Dunkard.....	69	382
.....	10	392
.....	12	404
.....	5	409
.....	11	420
.....	5	425
.....	20	445
.....	5	450
.....	15	465
.....	10	475
.....	5	480
.....	15	495
.....	20	515
.....	33	550
.....	25	575
.....	15	590
.....	42	632
ne.....	25	657
.....	15	672
.....	5	677
.....	20	697
.....	20	717
.....	15	732
.....	5	737
full of water.....	13	750
and.....	35	785
.....	25	810
.....	8	818
.....	82	900
.....	5	905
.....	20	925
.....	35	960
.....	10	970
.....	40	1,010
.....	23	1,033
.....	23	1,056
.....	15	1,071
.....	29	1,100
.....	40	1,140
njun (show of gas at 1,337 feet).....	264	1,404
.....	10	1,414
.....	12	1,426
.....	11	1,437
w.....	108	1,545
wells.....	160	1,705
r.....	20	1,725
.....	40	1,765
wells.....	15	1,780
.....	20	1,800
-foot.....	50	1,850
wells.....	40	1,890
wells.....	110	2,000
(Jordan).....	25	2,025
.....	15	2,040
.....	20	2,060
.....	10	2,070
.....	25	2,095
.....	20	2,115
.....	200	2,315
rd.....	15	2,330
.....	20	2,350
.....	25	2,375
beth.....	15	2,390
.....	20	2,410
.....	110	2,520
bells.....	78	2,598
wells.....	68	2,666

## DUNKARD TOWNSHIP.

Dunkard Township has been pretty thoroughly prospected, except in the northwest corner, and gas wells are numerous. There are 6 on the bank of Monongahela River between Dunkard Creek and the State line. These are on the Maple (36), Dilliner (241), Chisham (242), Sterling (243), and Sturgiss (244) farms. The Jennie Miller (245) and Stone (246) wells, on the hill above the Sturgiss farm, opposite Point Marion, are producing. A little over a mile east of Dunkard village there are over a dozen gas wells on the Durr, Keener, Watson, and Titus farms, which were drilled in 1904-5. All of these wells are on the steep flank of the Fayette anticline. Gas was found in the Myrtle Gabler well and in two or three others on the east and, structurally, higher side of the Garrison oil field. In the vicinity of Davistown and Bald Hill a number of wells are yielding good quantities of gas, and there are a few along the east side of the Mount Morris oil field where the anticlinal slope becomes steeper. A gas well (247) on the Hathaway farm, one-half mile north of the Valley House, at Davistown, was drilled by E. M. Hukill and plugged. It is said that the gas blew the plug out, and that when the well was closed a second time gas appeared in the coal mines at Davistown in such abundance that the miners had to stop work. Practically all the gas produced in this township comes from the Big Injun sand.

## GREENE TOWNSHIP.

Three wells in Greene Township have produced gas. One of these, the Hugh Keenan well (248), near Turkey Knob, struck a pocket of gas in the Big Injun sand. It was allowed to blow for several days, and each time it was tested the rock pressure fell off so much that the gas was never piped away. Two wells (249) on the Fordyce farm, on Frosty Run, came in fairly strong in 1905.

## JEFFERSON TOWNSHIP.

The first two wells in Jefferson Township were drilled at Rices Landing. One of these (250) was sunk in 1860 on the bank of Monongahela River, 50 feet from the mouth of Pumpkin Run, and got a little gas. Another well (251), drilled about the same time, at the old mill half a mile up the run, showed a sign of oil. Nothing is known of the depth of these wells or in what sands they found signs of oil, but it is said that neither paid for drilling.

This township lies along the east flank of the Bellevernon anticline and the western half, at least, should be good gas territory. Over 30 wells have been drilled here, with a very small percentage of failures. The Degood No. 2 (252) and Jacob Crayne (253) holes were dry

while wells on the Haver (254), Eaton (83), Denny (255), and Scott (85) farms yielded but little gas. A group of wells on the Luse (256), Degood (257), Horner (84), Sprowles (258), Robinson (259), and Williams (260) farms, at the north end of the township, has been described as part of the Clarksville field, much of which is in Washington County. Gas comes largely from the lower sands.

## MORGAN TOWNSHIP.

Between 50 and 60 wells have tested all parts of Morgan Township. So far as known, only 2 wells—the Cotterell (90), on Browns Run, and the Shape (92), at Castile—were abandoned when completed. The wells in this township are described in the Waynesburg folio (No. 121) as being in the Zollarsville, Clarksville, and Waynesburg fields, but developments of 1904-5 have filled in the intervening areas so that such discrimination is no longer possible. Near Clarksville the gas comes largely from the Gantz and Bayard sands. Elsewhere the Big Injun, Fifty-foot, and Fifth sands carry some gas, tho the largest flow comes from the Bayard sand. The axis of the Bellevernon anticline crosses the eastern part of the township, and from the structure and evidence of wells already drilled it seems that gas should be obtained almost anywhere except in the vicinity of Castile, an area which has proved to be barren. Drilling should not stop short of the Elizabeth sand, however, as many wells farther north in the Zollarsville field are strong producers from this sand. The Shape well (92), at Castile, 1 mile east of the Waynesburg synclinal axis, one of the deepest holes in Greene County, struck a strong flow of salt water in the Big Injun. The record of the John Bennett No. 1 well, drilled by the Carnegie Natural Gas Company in 1900, is given herewith:

*Record of John Bennett well No. 1 (89), Morgan Township.*

[Depths are to the top of the respective beds.]

	Thick- ness.	Depth.
	<i>Fect.</i>	<i>Fect.</i>
Coal, Mapletown.....		480
Coal, Pittsburg.....		580
Sand, Dunkard (show of oil at 1,080 feet).....	135	1,015
Sand, Salt (water at 1,480 feet).....		1,480
Sand, "Keenan" (?).....		1,570
Lime, Big.....		1,720
Sand, Big Injun.....		1,800
Sand, Fifty-foot.....		2,550
Sand, Gordon.....	20	2,735
Sand, Fourth.....	30	2,830
Sand, Fifth.....	30	2,890
Sand, Sixth or Bayard (gas at 3,020 feet).....	15	3,015

The gas in this part of the township is found mostly in the Fifty-foot and Bayard sands, altho in many wells the Big Injun and Fifth sands produce light flows.

## FRANKLIN TOWNSHIP.

More than 100 wells have been drilled in Franklin Township and about 10 per cent of these were failures. The Bellevernon anticline crosses the eastern part of the township and the Waynesburg syncline the western part. A larger number of the wells are in the northeast section of the township and extend from the crest of the anticline well down its western flank. The territory along the axis near the heads of Laurel and Sugar runs has been shunned because of two dry holes—the Inghram well (261), on Laurel Run, and the Wood well (262), on Smith Creek—evidence which seems hardly sufficient for condemning so large an area as is at present avoided.

Drilling for gas in Greene County was begun, so far as record show, at Waynesburg in 1885, when a well (201) was sunk by the Waynesburg Gas Company on the north bank of Tenmile Creek, at the mouth of Purmans Run. The record of this well was published in the Annual Report of the Geological Survey of Pennsylvania for 1886, part 2, page 772. The well, although favorably located, was not successful, for while drilling in a promising sand, with a show of oil at a depth of 2,745 feet, the cable parted; the drillers were unable to regain the tools and the hole was abandoned. Subsequent drilling in the immediate vicinity shows that if the well had been sunk a few feet deeper a strong flow of gas would, without doubt, have been struck. As it was, several years past before gas was found in paying quantities in this locality.

Probably the first producing well (263) in this field was drilled in April, 1889, on the Grimes farm, on Grimes Run,  $1\frac{1}{2}$  miles above its mouth and 2 miles northeast of Waynesburg. It is 2,900 feet deep and is still yielding a small amount of gas. A well (202) drilled on the Robert A. Sayers farm in February, 1889, at the site of the Carnegie Gas Company's pump station, 2 miles east of Waynesburg reached a depth of 2,675 feet without producing gas in paying quantity. The records of the Grimes and Sayers wells were published by the Second Geological Survey of Pennsylvania in Report I 5 1890, pages 312, 313.

A portion of this district is sometimes spoken of as the Bayard field, taking its name from the well (167) on the Thomas Bayard farm, Whiteley Township, where the Sixth or Bayard sand was discovered in February, 1895, at a depth of 2,400 feet below the Pittsburgh coal. This sand is the largest producer in the Waynesburg field. The average distance between the Pittsburgh coal and the Bayard sand is 2,433 feet.

Active drilling in the immediate vicinity of Waynesburg followed the discovery of gas on the E. M. Sayers farm (56), just west of the Waynesburg and Washington Railroad station. This well was sunk

in the flood plain of the creek and reached the Bayard sand at a depth of 2,945 feet below the surface. It was drilled by the Manufacturers' Light and Heat Company, Reese & Heasley, contractors, and was completed in February, 1900. The following is the record:

*Record of E. M. Sayers well (56), Waynesburg.*

	Thick- ness.	Depth.
	<i>Feet.</i>	<i>Feet.</i>
Conductor.....	16	16
Slate.....	79	95
Coal.....	3	98
Slate and lime.....	76	174
Coal, Waynesburg.....	3	177
Slate and lime.....	216	393
Coal, Mapletown.....	4	397
Lime.....	97	494
Coal, Pittsburg.....	6	500
Slate, red rock, and shells.....	575	1,075
Sand, Dunkard.....	50	1,125
Slate and shells.....	130	1,255
Sand.....	30	1,285
Slate.....	165	1,450
Sand, Salt.....	75	1,525
Slate, red rock, and shells.....	144	1,669
Lime, Big.....	43	1,712
Sand, Big Injun.....	298	2,010
Slate and shells.....	290	2,300
Sand, Thirty-foot.....	30	2,330
Slate and shells.....	100	2,430
Sand, Gantz, and Fifty-foot combined.....	55	2,485
Slate and sand shells.....	30	2,515
Red rock.....	10	2,525
Sand, Gordon Stray.....	25	2,550
Slate.....	2	2,552
Sand, red.....	20	2,572
Red rock.....	5	2,577
Sand, hard, and shells.....	10	2,587
Shells, red.....	43	2,630
Sand, pebble.....	36	2,666
Red rock.....	3	2,669
Slate, white.....	5	2,674
Shells, red.....	10	2,684
Sand, hard.....	20	2,704
Red rock.....	3	2,707
Sand, red.....	18	2,725
Sand, pebble.....	19	2,744
Sand, shells.....	30	2,774
Sand, gray.....	10	2,784
Slate.....	10	2,794
Sand, Fifth.....	44	2,838
Slate, black.....	89	2,927
Sand, Bayard.....	18	2,945

The most recent and extensive development in Franklin Township has been the drilling of a group of wells at the head of Smith Creek. These wells are close to the crest of the anticline and all but one have given good quantities of gas when completed. They are mostly Fifth sand wells. Many of the wells in this township get small quantities of gas in the Big Injun, Gantz, Gordon, and Fifth sands, but the Bayard is the greatest producer. Several holes west of Waynesburg are dry, and one, the Wisecarver well (65), on Wisecarver Run, got a flow of very salty water in the Bayard sand, which filled the hole, 1,200 feet, in three hours.



## WHITELEY TOWNSHIP.

Fourteen wells in the northern and western parts of Whiteley Township are yielding gas and 3 are dry. The southeastern half of the township is so far down in the Whiteley syncline that it probably is barren, as is shown by the dry holes on the Stevens (264) and Bov (265) farms. The Thomas Bayard well (167), in which a deep sand lying below the Fifth and now known as the Bayard sand was covered, is on Mount Phoebe Run in this township. The large productive field about Waynesburg has been developed since disclosure of a new source of gas by the drilling of this well in February, 1895. The distance from the Pittsburg coal to the top of Bayard sand in the original well is 2,410 feet.

Two wells on the Iseminger farm, although lying between producing wells less than half a mile apart, were dry. No. 1 (266) yielded so very little gas that it was abandoned when completed and No. 2 (267) was wholly barren. This may be due to the density of the reservoir rock. The record of the Josephus Bowers well (265) is detailed and is given here with correlations. This well is on Lantz Run, three-fourths of a mile above Whiteley Creek.

*Record of Josephus Bowers well (265), Whiteley Township.*

	Thick- ness.	D
	<i>Feet.</i>	
Coal, Pittsburg (top).....	120	
Sand, Salt.....	235	
Lime, Big (top).....	40	
Sand, Big Injun.....	60	
Slate.....	15	
Slate and shells.....	20	
Sand.....	8	
Slate.....	47	
Slate and shells.....	30	
Sand.....	25	
Slate.....	25	
Sand, Thirty-foot.....	75	
Slate and shells.....	10	
Sand.....	15	
Slate.....	20	
Sand, Gantz.....	60	
Slate and shells.....	30	
Sand.....	20	
Slate.....	15	
Sand.....	40	
Red rock.....	15	
Slate.....	12	
Sand, dark.....	40	
Red rock.....	10	
Sand, Gordon.....	80	
Red rock and shale.....	18	
Sand.....	40	
Slate and shale.....	50	
Red rock and shale.....	10	
Sand, dark, Fifth.....	45	
Slate and shale.....	6	
Sand, white.....	29	
Slate.....	15	
Sand, Bayard (dark).....	50	
Slate and shale.....	2	
Shell (gas).....	130	
Slate.....		

## PERRY TOWNSHIP.

On the eastern side of the Mount Morris oil pool gas has been obtained in several wells, including those on the Bowen (268), Evans (269), Ryan (270), and Gump (271) farms. About 6 wells have been drilled west of Mount Morris. The Williamson well (272), on Shannon Run, about a mile above its mouth, is said to have shown a little oil in the Fifth sand, while the Delaney (105) well, just below the mouth of Hobbs Run, has been supplying gas to Newtown for a number of years. A well (228) on the Headley heirs' farm, drilled about 1890, is said to have yielded a strong flow of oil at first, but when the well was shot the oil was drowned by a rush of salt water. The Samuel Minor well (273), at the head of Hackelbender Run, produced a little gas from the Big Injun, but was abandoned when completed. This line of wells across the township seems to be considered as conclusive proof of the barrenness of the sands between Mount Morris and Rudolph Run, but the Shriver and Brown oil wells at the mouth of Blacks Run, a short distance south of the State line, and the report of salt water and oil in the Headley well (228) suggest that a small pool might be found by drilling on the ridge east of Blacks Run or on Little Shannon Run about one-fourth of a mile below the Headley well.

## WASHINGTON TOWNSHIP.

Less than 20 wells have been drilled in Washington Township to date. Several of these in the Fonner field west of Hope produce oil. Two wells on the Closser farm (147), on Overflowing Run, found gas, but a third was dry. The Conger well (148), at the mouth of Overflowing Run, is a light "gasser," as is also the Allison well (146), on Wisecarver Run. At Ruff Creek post-office a well (226) drilled on the Ellen Ross farm gave at first about a barrel of oil a day and a little gas from the Gantz sand, but it has been considered a dry well.

Dry holes were completed on the Hoge (149), Hoffman (274), and Stilwell (151) farms. All the evidence shows that the township as a whole may be considered rather barren of either oil or gas, a condition which seems to be explained by the course of the Waynesburg syncline and the low dip of the rocks, together with the absence of water in the deep sands.

## WAYNE TOWNSHIP.

About 120 deep wells have been drilled in Wayne Township; of this number, 30 are oil wells, 75 gas wells, and 13 unproductive. The group of oil wells described above as the Lantz field is located near the mouth of Hoovers Run and extends southward into West Virginia.

The gas wells are scattered over the northern-central part of the township and thickly distributed along its western side in the Wayneburg syncline. Most of these wells have been drilled since the summer of 1902, but the first well in the Lantz oil field was drilled in 1899.

To a group of wells around Kneisley schoolhouse at the head Pursley Creek the name Kneisley field is given. All the wells in the field were completed in the Fifth sand with a good flow of gas, except the Cornelison (275), which was dry in the Fifth sand but struck a good flow of gas in the Bayard sand. The Guthrie No. 1 (27) showed a gas pressure of 810 pounds in ten minutes and a rock pressure of 865 pounds. The Arthur Hoy well (156) was completed at a depth of 3,492 feet, with a big flow of gas from the Fifth sand. It was drilled by the Carnegie Natural Gas Company in 1903, and its record is as follows:

*Record of Arthur Hoy well (156), Wayne township.*

	Thick- ness.	Dep
	Feet.	Feet.
Coal, Mapletown.....	5	1.
Coal, Pittsburg.....	8	1.
Slate and red rock (water at 1,472 feet).....	365	1.
Sand.....	16	1.
Slate.....	45	1.
Sand, Big Dunkard.....	40	1.
Slate and shells.....	300	1.
Sand, gas.....	30	2.
Slate.....	40	2.
Sand, Salt (hole full of water at 2,072 feet; water at 2,145 feet).....	150	2.
Slate, shells, and lime.....	110	2.
Lime, Big.....	57	2.
Sand, Big Injun (some gas at 2,397 feet).....	265	2.
Sand, Thirty-foot.....	32	2.
Sand, Gantz.....	5	3.
Sand, Fifty-foot.....	100	3.
Sand, Gordon Stray.....	20	3.
Sand, Gordon.....	10	3.
Sand, Fourth.....	20	3.
Sand, Stray.....	25	3.
Sand, Fifth (little gas at 3,453 feet; big gas at 3,460 feet).....		3.
Total depth (completed in black sand).....		3,492

On Roberts Run, between Spraggs and Blacksville, 8 or 10 wells have been drilled. All seem to be near the anticlinal axis, but the fold is low, disappearing to the south. About half of these wells produced strong flows of gas when they were completed, while others were so weak that they were abandoned. A number of companies have been engaged in the attempt to develop a field in this region, some looking for gas and others for oil, but without marked success. A small quantity of oil was found in the Big Injun sand in a well (162) on the H. M. Spragg farm. The Worley well (277), 1 mile northeast of Blacksville, is said to have been one of the biggest gas producers in Greene County. The Pittsburg coal was found higher in this well than in those around it, and the fact that all the others in the vicinity except the Brock No. 1 (278) were dry led the drillers to believe that the gas belt was "so narrow it could be cut in two by a 10-inch bit."

There is some evidence to indicate that this well is at the north end of an anticline which crosses the State line just east of Blacksville.

South of the Kneisley group of wells the field in western Wayne Township generally goes by the name of the Kuhntown or Hoovers Run field. Most of the wells in this belt lie on the east side of the syncline, but Tustin heirs' well (279) is west of the axis and the H. L. Granlee well (280) occupies a position almost exactly in the center of the basin. South of Blockhouse Run the field bends westward and extends over a mile across the synclinal axis in Gilmore Township. At Pennsylvania Fork of Dunkard Creek the breadth of the producing area from east to west is  $2\frac{1}{2}$  miles. In general the eastern edge of this field as at present developed can be said to lie one-half mile southeast of Hoovers Run village and to cross Pennsylvania Fork at the mouth of Toms Run. Three gas wells, namely, the T. E. Eddy (281), Henderson No. 2 (282), and Jacob Eddy No. 2 (283), lie on a line one-half mile east of this, and the last two of these are approximately on the western border of the oil field. Between the oil and gas fields is a gap of more than half a mile in which no wells are known to have been drilled.

A dry hole (204) drilled in 1900 on the Lewis Kuhn heirs' farm, in a side valley 1 mile southeast of Hoovers Run village, reached a depth of 3,780 feet below the surface, or over 500 feet below the horizon of the Elizabeth sand, without striking gas in any quantity. As this is the second deepest hole in Greene County the record is given here.

*Record of Lewis Kuhn heirs' well (204), Wayne township.*

	Thick- ness.	Depth.
	Feet.	Feet.
Coal, Pittsburg.....		750
Sand, Dunkard.....	30	1,150
Sand, Salt (salt water at 1,628 feet).....	147	1,767
Sand, Big Injun.....	174	2,200
Sand, Fifty-foot.....	58	2,774
Sand, Gordon.....	15	2,946
Sand, Fifth (?), hard.....	50	3,098
Sand, limy, hard.....	300	3,550
Red rock, soft.....	5	3,555
Lime, sandy.....	20	3,575
Slate, soft.....	15	3,590
Lime, sandy.....	60	3,650
Sand, white (Speechley?).....	15	3,665
Slate, soft.....	10	3,675
Lime, sandy.....	105	3,780

Gas at 2,026, 2,985, 3,052, and 3,062 feet; oil in Dunkard sand.

At the time the Kuhn well was drilled the sands had not been definitely identified in this field, and consequently few details were recorded, and even the position of the Fifth sand is doubtful. The record of the Felix Bell No. 1 well (152),<sup>a</sup> 1.7 miles south of Hoovers Run village, is exceptionally good, and is given on the next page.

<sup>a</sup> *West Virginia Geol. Survey*, vol. 1 (a), 1904, p. 122.

*Record of Felix Bell No. 1 well (152), Wayne Township.*

	Thick- ness.	D
	<i>Fet.</i>	<i>1</i>
Conductor.....	9	
Unrecorded.....	266	
Sand, Bluff (Waynesburg) (water).....	45	
Coal, Mount Morris (Waynesburg) (top).....		
Unrecorded.....	250	
Coal, Mapletown (Sewickley) (top).....		
Unrecorded.....	95	
Coal, Pittsburg (top).....		
Unrecorded (10-inch casing at 685 feet).....	20	
Lime and slate.....	90	
Red cave.....	10	
Slate and shells.....	90	
Sand, dark.....	25	
Slate.....	20	
Red cave.....	40	
Slate.....	30	
Sand, dark gray.....	15	
Coal.....		
Slate.....	35	
Red cave.....	20	
Slate and shell.....	95	
Red cave.....	10	
Sand, gray.....	55	
Coal (Upper Freeport).....		
Slate.....	25	
Sand, light gray.....	130	
Slate and shell.....	25	
Sand, light gray.....	50	
Slate and lime.....	25	
Sand, white.....	70	
Slate.....	45	
Sand, white.....	25	
Slate.....	10	
Sand and lime, black.....	20	
Sand, white.....	30	
Slate.....	5	
Sand, dark gray.....	70	
Red cave.....	70	
Slate and shell.....	40	
Lime, Big lime.....	65	
Sand, Keener (gas).....	20	
Sand, Big Injun, white and gray.....	50	
Slate.....	20	
Sand, gray (bottom of Big Injun).....	70	
Slate.....	5	
Sand, gray.....	110	
Slate and shell.....	90	
Sand, black.....	5	
Slate and shell.....	15	
Sand, dark gray.....	35	
Slate and shell.....	130	
Sand, gray (Fifty-foot).....	25	
Slate.....	5	
Sand, brown.....	8	
Slate.....	4	
Sand, pink.....	18	
Shelly.....	45	
Sand, gray (Stray).....	30	
Slate and shell.....	10	
Sand, gray.....10		
Slate.....3 Gordon.....	35	
Sand, white.....22		
Slate and shell.....	20	
Sand, dark.....	5	
Slate and shell.....	20	
Sand, brown.....	5	
Slate and shell.....	10	
Sand, pink.....	5	
Slate.....	2	
Sand, dark gray (Fourth) (gas).....	13	
Slate and shells.....	29	
Shells.....	6	
Sand, dark.....	10	
Slate, red, and shells.....	17	
Sand, gray.....10		
Slate.....2		
Sand, dark.....12		
Slate.....10 McDonald or Fifth sand (gas at 3,001 feet).....	61	
Sand, dark.....3		
Slate.....3		
Sand (small pebbles).....15		

In this well and in most wells throughout the Hoovers Run field the greatest amount of gas is produced from the Fifth sand, although a number of wells obtain large supplies from the Big Injun and small amounts are found by two wells in the Fifty-foot sand and by several in the Gordon and Fourth sands. As the Fifth sand is the principal producing bed in this township, it is sometimes called the Gas sand.

#### MORRIS TOWNSHIP.

More oil wells and more dry holes have been drilled in Morris Township than gas wells. Three wells at Simpson Store and 4 wells about one-half mile northwest of Deerlick were producing gas at the time this region was last visited. Four other wells scattered over the area are said to have produced small quantities. These are the James Iams wells (284), on Bates Fork midway between Deerlick and Swarts; the Samuel Phillips well (285), on Browns Fork,  $2\frac{1}{2}$  miles southeast of Nineveh; one well near the western edge of the Nineveh oil field; and the Laban L. Gray well (286), 1.4 miles east of Deerlick. On account of the wild-cat nature of these wells and consequent lack of available records, it is not possible to give any definite information regarding them, but the production is believed to have been small. Near the west end of the Fonnér oil field a number of dry holes have been drilled, which serve to define its limits in this direction. Other dry holes were drilled south of the Nineveh oil field in a vain endeavor to enlarge the productive territory. In 1903 a dry hole (97) was drilled on the James Dunn farm, midway between the Fonnér field and the gas field northwest of Deerlick. This well passed through the Bayard sand, which was unproductive. At Deerlick, 1.2 miles southeast of the Dunn well, a dry hole was drilled in 1895 through the same bed. Two miles farther south-southeast there is a dry hole (103) on the Hugh Simpson farm, 1 mile southwest of Swarts. This hole passed through the Fifth sand. The Samuel Phillips well (285), mentioned above, is  $1\frac{1}{2}$  miles south of the Simpson well and is said to have produced a little gas. Another dry hole was drilled in 1889 on the Hugh Auld farm (94), 1.7 miles northwest of Nineveh. This hole is said to have reached the Gordon and to have found some gas and much salt water. West of the Nineveh field dry holes have been drilled on the John Parkinson farm (326), 1.6 miles northeast of Time, and on the James Carl farm (287), 1 mile farther north.

The following record of the John Lewis well (206) is given to show the general character of the section:

*Record of John Lewis well No. 1 (206), Morris Township.*

	Thick- ness.	Depth.
	<i>Feet.</i>	<i>Feet.</i>
Clay, yellow, soft.....	16	16
Shale, black.....	164	180
Sand, white.....	15	195
Shale, black.....	105	300
Lime, white, hard.....	100	400
Shale, black, soft.....	150	550
Sand, white, hard.....	25	575
Shale, black, soft.....	25	600
Lime, white, hard.....	35	635
Coal, Waynesburg.....	5	640
Lime, white, hard.....	195	835
Coal, Mapletown.....	5	840
Lime, white, hard.....	95	935
Coal, Pittsburg.....	5	940
Lime, white, hard.....	100	1,045
Shale, red, soft.....	55	1,100
Lime, white, hard.....	100	1,200
Shale, black, soft.....	135	1,335
Sand, Little Dunkard.....	15	1,350
Shale, black, soft.....	100	1,450
Sand, Big Dunkard, white, hard.....	60	1,510
Shale, white, soft.....	50	1,560
Sand, white, hard.....	25	1,585
Shale, black, soft.....	100	1,685
Sand, gas, soft and very open.....	85	1,770
Shale, black, soft.....	32	1,802
Sand, salt, white, hard.....	108	1,910
Shale, black, soft.....	170	2,080
Lime (Big lime), white, hard.....	55	2,135
Sand, Big Injun, white, hard.....	250	2,385
Shale, white, soft.....	320	2,705
Sand, Thirty-foot, white, hard.....	30	2,735
Shale, black, soft.....	115	2,850
Sand, Fifty-foot, white, hard.....	50	2,900
Shale, white, soft.....	30	2,930
Sand, Nineveh, white, hard (gas at 2,940 feet).....	35	2,965

CENTER TOWNSHIP.

A dozen or more wells on Stewart Run and Pursley Creek below Oak Forest constitute the only gas field in this large township. Most of the wells have been drilled since 1903 and, although low in the syncline, are giving fair quantities of gas from the lower sands. A few other scattering wells are producing gas. These include the James Adamson well (288) and the W. M. Boler well (289), 0.7 mile and 1.3 miles, respectively, east of Rogersville. Several wells in various parts of the township, namely, the A. R. White well (290) near Bluff, the B. L. Woodruff No. 1 (291) at Woodruff, the Thomas Grove well (24) in a ravine on Hargus Creek 1.4 miles east of Woodruff, and one or more wells in the Grays Fork field, are said to have produced some gas, but at present they are unproductive. The following is a record of the Thomas Grove well:

*Record of Thomas Grove well (24), Center Township.*

	Thick- ness.	Depth.
	Feet.	Feet.
Coal, Waynesburg.....		335
Coal, Mapletown.....		625
Red cave.....	115	1,115
Sand, Gas.....		1,500
Sand, Salt (salt water at 1,620 feet; break in Salt sand at 1,650 feet).....	216	1,789
Slate and red slate.....	74	1,863
Lime, Big.....	75	1,938
Sand, Big Injun (break at 2,100 feet).....	284	2,222
Slate.....	96	2,320
Sand, Squaw.....	80	2,400
Slate and lime.....	108	2,508
Sand, Thirty-foot.....	62	2,570
Slate and shells.....	140	2,710
Sand, Fifty-foot (gas and show of oil at 2,715 feet).....	76	2,786
Slate.....	5	2,791
Shells and sand.....	33	2,824
Sand, gray (Nineveh).....	11	2,835
Slate and shells, red rock.....	30	2,865
"Pencil cave".....	5	2,870
Sand, Gordon (gas at 2,876 feet).....	37	2,907
Slate and shells.....	18	2,925
Red rock.....	57	2,982
Sand, Fourth.....	23	3,005
Sand, hard, shells and slate.....	45	3,050
Sand, Fifth.....	35	3,085
Slate.....	105	3,190

Most of the dry holes are located in the northwest corner of the township, on a continuation of the belt of holes extending between the Nineveh and Bristoria oil fields. East of the Nineveh syncline wells have been drilled for oil on the Ross heirs' farm (292), 0.6 mile north of Rutan, and on the Scott farm (28), 1 mile southwest of Rutan, but both were dry. On the structural slope rising toward the Amity anticline no wells have been drilled for about 2 miles; but near the head of Clover Run an unsuccessful attempt was once made for oil on the A. I. Lindsey farm (293).

## JACKSON TOWNSHIP.

In the vicinity of Delphene, on the southeastern border of the Bristoria oil field and extending up the anticlinal slope for over a mile, lies a field of about a dozen wells which produce gas mainly from the Gordon, Gordon Stray, and Fourth sands. As can be seen from the structure contours the relation of this field to the adjacent oil field is normal. All the wells at present producing gas lie south-east of a line passing in a northeast-southwest direction thru the mouth of the run entering Job Creek below the store at Delphene. The wells west of this line in the township once produced gas, but are now abandoned. At least 4 wells—the J. L. Pethtel No. 2 (78), L. C. Johnson No. 2 (294), and Karl Crawford Nos. 1 (295) and 2 (296)—in the Bristoria oil field have produced gas. In these scattering wells the gas is derived from the Gordon and Gordon Stray sands and is found only in cases where the drill passes below the producing oil sand.



Outside the Delphene and Bristoria fields, gas has been produced in two wells higher up on the anticline. These are the A. C. Fordyce well (297), about 1 mile northwest of Bluff, and the Elijah Maple well (298), 2.8 miles south of Delphene. A little gas is reported in 2 wells in the vicinity of Woodruff and in 1 well  $1\frac{1}{2}$  miles southeast of Delphene. The Hughes No. 1 well (73), at White Cottage was dry. The only part of this township which has been thoroughly tested for oil or gas is the northwest corner, and in the entire area east and southeast of Delphene only 7 holes are known to have been drilled. In a strip  $2\frac{1}{2}$  miles wide crossing the southern side of the township, so far as known, only 1 hole has been put down.

#### GILMORE TOWNSHIP.

The Hoovers Run field, which lies mainly in Wayne Township, veers to the west into the southeast corner of Gilmore Township as far as Jollytown, near which about a dozen wells are now producing gas. The principal producing sands in this field are the Fifth and Big Injun.

Gas has been found in the Garrison well (67), on Garrison Fork,  $1\frac{1}{2}$  miles west of Jollytown, and in the J. P. Hagan well (299), 2 miles west of Jollytown, near the mouth of Six Run. The J. B. Fordyce well (66), 2 miles west of Pinebank, and the Josephus Rice well (300), 2 miles east of New Freeport, also produce gas. There is a dry hole on Blockhouse Run 1 mile below Triumph and another on Garrison Fork three-fourths of a mile above Bloody Run. The areas between Garrison Fork and Ashtree and that west of Six Run had not been tested by the drill up to the time this region was visited.

In view of the peculiar relations of the Hoovers Run field to the geologic structure, the probability of the further extension of the field in this township seems almost hopeless. However, an anticlinal axis crosses the township less than a mile west of Pinebank and Jollytown, and as gas is most likely to be found on anticlinal folds, it would seem worth while to test rather thoroly the territory in the vicinity of this axis. West of Jollytown and Pinebank only six wells have been drilled in the township, and as four of these have found gas there would seem to be a good chance of profit in prospecting the area.

#### RICHHILL TOWNSHIP.

The largest producing gas area in western Greene County is the Richhill field, which covers most of the northern-central part of this township. This field occupies the broad, triangular, knob-like summit of the Washington anticline. Gas is found at a number of horizons, including those of the Salt, Big Injun, Nineveh, Gordon Stray, Gordon, and Fourth sands. Owing to its occurrence in the

upper sands, gas is generally used in drilling the lower portion of the well. The principal operations here are conducted by the Natural Gas Company of West Virginia. This field contains about 35 wells, including 2 wells on the boundary line of Morris Township near Simpson Store. One of the best sections in this field is given by the Orndorf No. 1 well (301), near Graysville:

*Record of Orndorf No. 1 well (301), Richhill Township.*

	Thick- ness.	Depth.
	Feet.	Feet.
Coal, Waynesburg.....		312
Coal, Mapletown.....		512
Coal, Pittsburg.....		618
First red rock.....	15	800
Second red rock.....	10	820
Coal.....	2	847
Red rock.....	40	925
Slate, white.....	20	945
Coal.....	3	995
Sand, Hurry up.....	75	1,188
Slate, soft.....	10	1,390
Sand, Gas.....		1,480
Sand, Salt, containing a little coal.....	125	1,600
Lime, Big lime.....	45	1,790
Sand, Big Injun (some bad-smelling oil at 1,890 feet).....	253	2,043
Sand, Thirty-foot.....	30	2,405
Sand, Cantz.....	15	2,510
Sand, Fifty-foot (considerable gas at 2,572 feet; bottom of first streak of sand at 2,581 feet).....	30	2,599
Sand, Nineveh Thirty-foot, red (no sign of oil).....	13	2,682
Cave above the Gordon.....	37	2,719
Sand, Gordon Stray (oil at 2,737 feet; slate at 2,743 feet).....	31	2,750
Sand, Gordon.....	8	2,777
Sand, Fourth.....	10	2,814
Sand, Fifth.....	10	2,872

Outside the Richhill field natural gas is widely distributed in this township. Near the junction of Long Run and Job Creek and between that place and the mouth of Wright Run are six wells, producing gas from various sands. In the valley of South Fork, within  $1\frac{1}{2}$  miles of McCracken, are situated several gas wells, and to this small group the name Sugar Grove field has sometimes been applied, after an old name for McCracken. The first wells obtained gas in the Fifty-foot sand. Southeast of Jacksonville the crest of the Washington anticline is not known to have been tested. West of South Fork, within  $1\frac{1}{2}$  miles of the State line, there are five gas wells, proving the continuity of the Richhill field in this direction. In the southwest corner of Richhill Township the well records are generally very poor and therefore no further statement can be made regarding the producing sands. From what is known of the occurrence of gas elsewhere in this and in Aleppo Township it seems reasonable to suppose it may be found in almost any sand from the Salt down to the Fifth. This township has been fairly well tested both for gas and oil. The locations of dry holes, which are valuable as danger signals, are shown on the map. Several of these are located in the valley of Long Run in the extreme southeast corner of the township, near the

axis of the Nineveh syncline, in the northwest border of the Bristoria oil field. The absence of oil or gas may possibly be due to the local occurrence of salt water, but in this region salt water is generally absent. A well drilled through the Bayard sand, three-fourths of a mile north of Bristoria, was unproductive. In 1904 a well was drilled 1 mile northeast of McCracken and proved to be dry. Two attempts have been made to find gas and oil on the steep flank of the anticline between the Richhill and Grays Fork fields in the vicinity of Graysville, but without success.

#### ALEPPO TOWNSHIP.

Considerable gas has been found in various parts of Aleppo Township, but no well-defined fields of any size occur. At the southwest corner of the Bristoria oil field there are two gas wells which may be considered as the western extension of the Delphene field. Two dry holes have been drilled 1 mile and  $1\frac{1}{2}$  miles south-southwest of Higbee. Around the north end of the New Freeport oil pool there are several gas wells. With one exception these wells obtain gas in the Nineveh sand. The S. C. Leighliter No. 1 well (302) is productive in the Big Injun sand and did not go below that horizon. The John T. Elbin No. 2 (4) is producing both from the Big Injun and Nineveh sands. This well was drilled into the Fourth and obtained a show of oil in the Gordon. The following is its record:

*Record of J. T. Elbin No. 2 well (4), Aleppo Township.*

	Thick- ness.	Depth.
	<i>Feet.</i>	<i>Feet.</i>
Coal, Pittsburg.....		920
Sand, Salt.....	80	1,000
Line, Big line.....	70	2,193
Sand, Big Injun (first gas at 2,193 feet; second gas at 2,283 feet).....	254	2,447
Sand, Thirty-foot.....	25	2,800
Sand, Fifty-foot.....	45	2,905
Sand, Nineveh (gas at 3,029 feet).....	18	3,036
Sand, Stray.....	45	3,088
Sand, Gordon (show of oil at 3,129 feet).....	20	3,140
Sand, Fourth.....	12	3,209
Total depth.....		3,263

A dry hole (303) was drilled on the Robert Ullom farm, 2 miles southeast of Aleppo and 0.3 mile east of the Morris L. Pethtel No. 1 (17) well. This small group of wells and also the two wells mentioned above as forming the west end of the Delphene field lie on a westward projection of the structure, between the Bristoria and New Freeport oil fields. West of the north end of the New Freeport oil field, lying lower down the synclinal slope than most of the oil wells, is a group of four gas wells. One of these wells (304), on the Elizabeth Sammons farm, obtained gas in the Big Injun sand and went no deeper. A second well on this farm obtained gas in the Nineveh and Gordon

sands, in addition to a small show of oil in the Gordon. The westernmost of these four wells—the Christ. Grim No. 1 (7)—has been abandoned, but the record shows that it found gas in the Big Injun and Fourth sands and oil in the Gordon. A well (305) drilled in 1897 on the Anderson Hinerman farm,  $1\frac{1}{2}$  miles east of Morford, was abandoned, having found nothing but a small show of oil in the Gordon sand.

In this township there are a few scattering gas wells west of the Nineveh syncline. Several of them lie just outside the Aleppo oil field and on all sides except its lower or synclinal side. These are the Lewis Kuhn (306), John Moore (307), John Riggle (308), Clara Riggle (309), and Rachel Grim (310) wells, all of which are within a mile of Aleppo. The Rachel Grim well produces both gas and oil, the gas being derived from the Big Injun and Nineveh sands and the oil from the Fourth. The rock pressure of this well was 1,380 pounds. Gas is produced from several horizons in these wells, but the greater part of it probably comes from the Nineveh sand. The record of the Clara Riggle well (309), which obtained gas in the Bayard sand, is given below:

*Record of Clara Riggle well (309), Aleppo Township.*

	Thick- ness.	Depth.
	<i>Fect.</i>	<i>Fect.</i>
Coal, Mapletown.....		895
Coal, Pittsburg.....		997
Sand, Murphy.....	45	1,155
Sand, Dunkard.....	60	1,445
Sand, Gas.....	65	1,810
Sand, Salt.....	110	2,010
Lime.....	80	2,230
Sand, Big Injun.....	270	2,500
Sand, Gantz (top).....		2,770
Sand, Fifty-foot (gas at 2,906 feet).....	37	3,030
Sand, Nineveh (gas at 3,065 feet).....	15	3,075
Red rock.....	3	3,078
Sand, Gordon Stray.....	39	3,117
Sand, Fourth.....	13	3,221
Sand, Bayard (top) (gas).....		3,410
Total depth.....		3,447

Along the run between Aleppo and Morford there are 3 dry holes, and another lies half a mile west of Morford. These 4 holes lie very nearly along the strike and close to the synclinal axis. They penetrated the Gordon or the Fourth sand.

About 1 mile northwest of the Aleppo field a dry hole (19) was drilled on the J. E. Staggers farm, but it penetrated only a short distance beyond the Nineveh sand, in which a show of gas was found. Across the road northwest of this well is a gas well (311) on the Charles Bean farm. As can be seen from the map, the contours indicate something of a structural flat here. From the best evidence obtainable the Staggers well seems to be situated on the sharp edge of this bench and the Bean well a short distance west of

the edge. It seems possible that further drilling on this bench may obtain more gas. At a point 0.4 mile northeast of the Staggers well, or 1.1 miles northwest of Aleppo, a dry hole was once drilled on the Grimes farm. This well has about the same structural relations as the Staggers well, but is a little farther down in the syncline. Along the strike to the northeast, however, is a well (312) on the Thomas Thorpe farm, 1.7 miles north of Aleppo, on Blacks Creek, where gas is said to have been obtained. Gas is said to occur also in the Levi Thorpe well (313), 0.6 mile farther west on the same creek; the John Lewis well (314), on South Fork at the Richhill Township line; the M. L. Polen well (315), on Mudlick Fork, 0.8 mile south of McCracken; the Anderson well (316), on a tributary to Mudlick Fork, one-half mile southwest of the Polen well, and the Benjamin Phillips well (317), on Hewitt Run near the State line. On a tributary to Mudlick Run, 0.9 mile west-southwest of the Anderson well and 1.8 miles southwest of McCracken, a well (203) was drilled in 1904 on the Benson heirs' farm to a depth of over 5,300 feet, but was unproductive. Lying south of the Benson well and northwest of the line of dry holes extending from a point near Morford to the vicinity of Aleppo is an area of about 5 square miles which is not known to have been tested; but as its structural relations are similar to those of the Aleppo field, it may be expected to yield some oil or gas.

#### SPRINGHILL TOWNSHIP.

The New Freeport gas field covers a small area lying east of the oil field of the same name and extending southward from New Freeport. This field contains about 15 productive wells. Gas is produced from the Nineveh, Gordon, Fourth, and Fifth sands. According to report the rock pressure in one well in the Gordon was as high as 1,000 pounds, but in other instances it is less, being generally 500 to 550 pounds. This field lies on the east slope of a minor anticlinal fold which enters Greene County from the south. At present the field extends less than 2 miles south of New Freeport, but it seems possible that further drilling may enlarge it in this direction. It is also possible that other productive wells will be found northeast of New Freeport, as a test well about a mile northeast of that village is said to have found gas. Gas is also produced in the A. J. Dye well (318), 1.4 miles north of New Freeport and half a mile east of the productive oil belt. It would seem worth while to test the territory between these 2 wells and also farther northeast along the strike in the direction of Nettle Hill.

Another group of gas wells lies near the crest of the small anticline between Deep Valley and the State line on the south. These wells are less than 10 in number and do not form a definite gas field, but are scattered among producing oil wells or along the west side of

the oil field. The gas is obtained mainly from the Salvation sand, but a few wells find it in the Gas, Salt, and Big Injun sands. The lack of oil in these wells is explained by the fact that they do not reach the Nineveh sand. When the supply of gas fails they will doubtless be drilled to a greater depth. Three miles southwest of Deep Valley, near the State line, a well (136) was drilled in 1903 on the D. R. Meigham farm, and gas was found in the Salvation sand. This well was drilled through the horizon of the Gordon sand for oil, but without success. It is probable that the gas field will be extended in this part of the township. The record of the Meigham well is as follows:

*Record of D. R. Meigham No. 1 well (136), Springhill Township.*

	Thick- ness.	Depth.
	<i>Fect.</i>	<i>Fect.</i>
Coal, Pittsburg.....	6	1,086
Sand, Big Dunkard.....	110	1,680
Sand, Gas.....	60	1,890
Lime (water at 1,960 feet; gas at 2,010 feet).....	25	1,970
Lime, Big.....	75	2,335
Sand, Big Injun.....	165	2,590
Sand, Fifty-foot.....	15	3,090
Sand, Nineveh.....	11	3,128
Sand and shells.....	7	3,140
Do.....	7	3,175
Do.....	9	3,195
Sand, with slate.....	5	3,243
Shells.....	5	3,295
Total depth.....		3,424

About two-thirds of the distance between the Meigham well and the Hutchinson well (131), in the Board Tree oil field, a dry hole (319) was once drilled on the J. L. Riggs farm, on Knob Run. Dry holes have been drilled on the Caseman farm (128), 1.2 miles west of Deep Valley, and on a farm (320) on Wagonroad Run, 1 mile north of Deep Valley. At least 1 well in the New Freeport oil field, near the northern border of the township, produces gas.

#### RELATION OF OIL AND GAS POOLS TO GEOLOGIC STRUCTURE.

From a study of the relation of oil and gas pools to the geologic structure shown on the accompanying map (Pl. I), certain general statements can be made in respect to their mode of occurrence. It will be seen by reference to the map that in this county gas is found most abundantly along the axes of the anticlines, but it also occurs on the steep flanks of the folds, on synclinal axes, and in the midst of oil pools. The location of oil pools in relation to structural features has been mentioned in the description of the fields. It has been shown that the Blackshire and Garrison pools are on the flank of an anticline along the line where there is a marked change in grade. The Dunkard Creek pool bears the same relation, accompanied by a short

and slight reverse dip, while the Willow Tree pool lies around a small basin. The Mount Morris pool, which is near the base of the western limb of the Fayette anticline and also at a point of decreased grade, is limited on the lower or western side by a thinning out of the porous pay streak. On the other hand, the Fonner oil field laps over an anticlinal axis, while the Nineveh and Bristoria fields cross a synclinal axis. From Bristoria southward to the State line there is an almost continuous belt of oil wells, the north end of which is in the bottom of the Nineveh syncline and the south end 175 feet higher, on a minor anticline.

As shown on the map, the Wright Run pool, which contains two oil wells midway between Bristoria and Jacksonville, lies on the steepest slope of the Washington anticline. This is a very unusual position for the accumulation of oil, and it seems probable that the pool is small.

The Lantz oil pool, in Wayne Township, is  $1\frac{1}{2}$  miles east of the syncline and separated from it by a gas pool, quite out of accord with the anticlinal theory.

Thus it is apparent that in Greene County there is not such a close agreement between the occurrence of oil and gas and the geologic structure as has been noted in other parts of the Appalachian basin. The anticlinal theory, of which so much has been written, does not seem to apply here, or at least it must be modified materially before it will account for the facts.

Since the ability to determine the location of new fields in advance of drilling and the most favorable line of extension of the existing fields depends on knowledge of the laws governing the flow of hydrocarbons through the rocks and of the conditions controlling the action of these laws, it is desirable to analyze somewhat closely the conditions which prevail in this area and to determine why it differs from others that are regarded as typical of the Appalachian region.

In undertaking such an analysis the first thing to be determined is the amount of water in the oil and gas bearing rocks—are they saturated, do they contain a little water, or are they dry? Unfortunately this is a sort of information that the driller rarely notes, unless the flow of water is phenomenal; consequently in old fields it is difficult to obtain reliable data on this point. Present knowledge regarding water in the rocks may be summarized as follows: The Salt sand is almost universally wet—so saturated with water as to fill up the hole and retard drilling. Fifty barrels an hour have been bailed from it without exhausting the flow. Water is found where the sand is soft. Where the Salt sand is very hard it may be dry. The Big Injun sand in some wells has a little very salty water near the top and bottom. At Mount Morris water occurs in a soft streak of rock a few feet below the oil pay streak and separated from it by dense, hard strata.

Water has been reported in widely scattered wells at various horizons, but the occurrences below the Salt sand are uncommon. A few wells in the Waynesburg syncline filled with water from the Bayard sand. In fact, some gas wells along the western edge of the Waynesburg field show a tendency to fill with water from the Bayard sand as the gas becomes exhausted. As a rule, however, water is not found in any abundance below the Salt sand, many wells being perfectly dry below that horizon.

In accounting for the accumulation of oil and gas it is therefore safe to assume that the rocks, at least below the Salt sand, are practically dry; but it is equally safe to say that they have not always been in this condition, as they originally consisted of water-laid material and so must have been thoroughly saturated. What caused the water to disappear and how it departed is a mystery, but in the lower rocks of Greene County it is practically all gone.

It is difficult to account for the circulation of hydrocarbons through dry rocks, but if the rocks were originally saturated is it necessary to suppose that the oil reached its present position since the rocks became dry? Is it not more reasonable to suppose that the accumulation of the oil dates back to the time when water was plentiful and that it played an all-important part in the movement of the oil? This being so, oil formed in the underlying shales would naturally be forced upward on the water as a medium until it reached the upper limit of the medium or encountered an impervious barrier. The amount of oil which will accumulate under such conditions depends largely on the texture of the rock in which it occurs. If the porous rock is a coarse sandstone, it will afford opportunity for the accumulation of a large quantity of oil; but if the texture is fine, the reservoir will hold only a small amount. If the impervious stratum is level, oil and gas will collect beneath it in a thin sheet having nowhere any great volume. If, however, the cap rock is inclined, oil and gas will be forced along beneath it until a reverse dip occurs or until the top of the water is reached. Here the oil will rest. Thus oil and gas from a considerable area may be collected in a belt or pool. To assume the existence of a continuous porous stratum is not wholly safe, however. The sand rocks are of varying degrees of porosity, being in some places composed of fine sand densely packed and in others made up of coarse pebbles. Also the sand grains may be loosely or tightly cemented together by silica, iron oxide, or calcite. In any case these differences in the porosity of the reservoir strata may produce results in the accumulation of gas and oil quite different from what they would have been were the rocks of uniform texture.

On the supposition that, whether the rocks be wet or dry, natural gas will rise in a porous stratum on account of its specific gravity,



which is less than that of water or air, until stopped by an impervious cover, then anticlinal crests, provided they are unbroken, are the best places to look for gas. Oil in water-bearing rocks that are tilted should be found accumulated on the crest of anticlines that are below water level and floating on the water at its upper limits. It can ascend through rocks only so far as they are saturated and must stop at water level.

The structural position of the oil pools and the dryness of the rocks in Greene County suggest the hypothesis that the oil accumulated in pools after the formation of the rocks, while they were yet saturated with water, but that with the recession or disappearance of the water the oil moved by gravity back down the slope of the bed to its present position. Under these conditions the oil would be stopped in its downward progress by a reversed dip, by a dip of lower angle, or by increased density or thinning out of the reservoir rock.

The position of the Dunkard Creek pool is believed to be due to a slight, short reversal of the dip. A structural bench across which gravity could not overcome friction holds the Blackshire and Garrison pools. Lessened grade and thinning out of the reservoir rock may account for the Mount Morris pool. The Lantz field, in Wayne Township, is distinctly higher structurally than the gas pool on the west. While there is a change in dip here which may explain the presence of the oil, it is possible also that lessened porosity retarded further downward movement of the oil, but did not prevent the large amount of gas in this region, by its expansive force, permeating the lower portion of the same sand. It does not seem impossible that where gas is formed in such abundance as to fill an anticline it may by its own abundance and expansion be forced into all those portions of the reservoir rock not occupied by oil. The oil pool from Nineveh to Deep Valley lies in the syncline, indicating the nonsaturation of the rocks.

The anticlinal theory holds good for the extension of pools where the rocks are saturated with water, but the points at which oil is likely to occur can not be judged in undeveloped territory until the structure has been accurately determined and the presence or absence of water in the rocks ascertained.

**PROSPECTIVE TERRITORY.**

In the preceding pages a few hints have been given in regard to further development of the gas and oil fields in this county. It is hazardous to make suggestions of this sort and they should be considered as only of speculative value. The writers are of the opinion that there are yet some areas of considerable extent which might well be tested. The occurrence of gas and oil can not be foretold, but the following localities are proposed for consideration:

An area of several square miles lying south of Waynesburg between Laurel Run and Smith Creek has been tested by two holes on the Inghram (261) and Wood (262) farms. These holes were dry, but this is not convincing evidence that gas may not be found on Sugar Run and on Laurel Run.

The Mount Morris pool possibly might be extended to Whiteley Creek by drilling just west of the 550-foot structure-contour line, as shown on the map (Pl. I). This opinion is based on the geologic structure, which swings to the northwest, on the position of the Litman well (325), and on the reported occurrence of oil (pp. 57-58) at the mouth of Woods Run. If the pool extends across Whiteley Creek, it must be narrow, for the Lantz well No. 2 (221), which is farthest down the dip, was dry and the Rice well (223), which is supposed to be near the upper limit of the pool, had only a small showing of oil.

Blacks Run or the ridge to the east, in Perry Township, is along the strike north from the Brown and Shriver oil wells, which are on Dunkard Creek just over the State line in West Virginia. The report that a strong flow of oil (p. 58) was obtained in the Headley well (228) on Little Shannon Run suggests the possibility that drilling on or near Blacks Run might result in the discovery of more oil.

Since many of the wells in the Dunkard Creek field were not exhausted but were abandoned because the flow of oil softened the rocks and the mud plugged the holes, it may be that considerable oil still remains in this pool. Testing the field would not be expensive, as the oil is only about 500 feet below the surface and either coal or gas for fuel can be obtained readily.

Except 6 wells on Grays Fork, a line of holes drilled on the west side of the Nineveh syncline, between Nineveh and Bristoria, proved unproductive, although the geologic structure suggests the possibility that there is a continuous pool of oil in this district. There is a chance that drilling along the axis or on the east side of the syncline between Nineveh and Delphene might be more successful. Only 2 holes, both of which were unproductive, have been drilled on the east side of the syncline between Nineveh and the head of Lick Run. This is hardly sufficient reason for condemning so large an area. It

would also seem desirable to prospect farther southwest and northwest of Deep Valley before those territories are finally given up as unproductive.

Other areas to test for oil lie between Higbee and the north end of the New Freeport field, just west of the group of gas wells, and between Aleppo and Bristoria west of the syncline. Between Morford and the Benson heirs' well (203) is a considerable area in which no tests are known to have been made. The same is true of the region between the Delphene and New Freeport gas fields, east of the oil belt. Throughout southern Jackson and northern Gilmore townships less than half a dozen holes have been put down and there is still probability of discovering pools of oil or gas. Gas should be found along the Washington anticline 1 mile east of Ryerson Station.

#### WELL RECORDS.

The advantage of keeping accurate and complete records is almost universally underestimated by the drillers. In the great majority of cases only one or two coal beds and the principal oil and gas horizons are noted. These records may answer the purpose for recognizing the sand, but tell nothing of the character or distribution of the intervening formations, and therefore it is impossible to draw from them any geologic conclusions of value.

A few drillers and contractors have kept very good records, in which the thickness and depth of all beds from the surface down to the very lowest have been noted. It is urged that more such records be kept. It is especially important to note the positions and character of all coal, red shale, and limestone beds, as on these the geologist depends most of all for his correlations. Such data frequently enable him to determine the limits of a certain formation and therefrom the geologic name corresponding to the driller's term for a certain sand. In keeping records care should be taken to distinguish between limestones and hard sandstones. Limestone can always be distinguished from other rocks by its effervescence when treated with a few drops of dilute hydrochloric acid.

To meet the needs of drillers and other persons who wish to keep pace with the most approved methods of taking notes, the United States Geological Survey keeps in stock pocket record books which may be obtained, without cost, by all who desire them and will furnish records to the Survey. The covers of these notebooks contain a few brief geologic notes and suggestions to drillers.

In the accompanying table abbreviated records of 170 wells in the county are given. These records have been chosen from several hundred by reason of their wide distribution and their completeness. The following abbreviations are used in the column headed "Owner:"

Carnegie.....	Carnegie Natural Gas Company.
South Penn.....	South Penn Oil Company.
Fort Pitt.....	Fort Pitt Gas Company.
N. G. W. V.....	Natural Gas Company of West Virginia.
Utility.....	Union Utility Company.
Greensboro.....	Greensboro Gas Company.
Ross.....	Tim Ross & Co.
Dunn.....	J. L. Dunn & Co.
A. O. D. C.....	American Oil Development Company.
Hukill.....	E. M. Hukill & Co.
D. O. & G.....	Dunkard Oil and Gas Company.
Wheeling.....	Wheeling Natural Gas Company.
West. Penn.....	Western Pennsylvania Natural Gas Company.
Mfrs. L. & H.....	Manufacturers Light and Heat Company.
Phila.....	Philadelphia Company.
Peoples.....	Peoples Natural Gas Company.
Monong.....	Monongahela Natural Gas Company.
Nin. Pet.....	Nineveh Petroleum Company.
Jefferson.....	Jefferson Oil and Gas Company.

In the column of elevations the letter B indicates that the elevation of the well mouth was obtained by barometer; L by spirit level.

Summarized record of

[Elevation in feet above

No. on Pl. 1.	Name of well.	Township.	Owner.	Eleva- tion.	Prod- uct.	Producing sand.
1	W. J. Bryan No. 5.....	Aleppo.....	South Penn.....	1,350 B.	Oil....	Nineveh.....
2	W. J. Bryan No. 11.....	do.....	do.....	1,506 L.	do.....	do.....
3	Wm. Clendenning No. 3.....	do.....	do.....	1,332 L.	do.....	do.....
4	J. T. Elbin No. 2.....	do.....	do.....	1,175 B.	Gas....	do.....
5	Phil Carey No. 1.....	do.....	Carnegie.....	1,060 B.	Oil....	Fourth.....
6	T. Z. Griffith No. 1.....	do.....	Tim Koss.....	1,202 B.	Dry....	do.....
7	Christ. Grim No. 1.....	do.....	do.....	1,165 B.	Gas....	Fourth.....
8	Elias Grim No. 1.....	do.....	Dunn.....	1,134 B.	Oil....	do.....
9	Wm. McQuay No. 3.....	do.....	South Penn.....	1,323 B.	do.....	Gordon.....
10	Jas. McVey No. 1.....	do.....	do.....	1,260 B.	Dry....	do.....
11	Jas. McVey No. 2.....	do.....	do.....	1,418 L.	Oil....	Nineveh.....
12	J. D. Miller No. 3.....	do.....	do.....	1,368 B.	do.....	do.....
13	Jacob Murray No. 1.....	do.....	Dunn.....	1,165 B.	do.....	do.....
14	Lewis Parry No. 1.....	do.....	South Penn.....	1,220 L.	Oil....	Nineveh.....
15	Z. T. Parry No. 1.....	do.....	do.....	1,261 L.	do.....	do.....
16	Peter Parson No. 2.....	do.....	do.....	1,462 L.	do.....	do.....
17	M. L. Pethtel No. 1.....	do.....	do.....	1,075 B.	do.....	do.....
18	Russell Sammons No. 2.....	do.....	do.....	1,390 B.	Oil....	Fourth.....
19	J. E. Staggers No. 1.....	do.....	do.....	1,219 L.	Dry....	do.....
20	Wm. Weimer No. 1.....	do.....	do.....	1,255 B.	Oil....	Fifth.....
21	L. G. Whipkey No. 1.....	do.....	do.....	1,332 L.	do.....	Nineveh.....
22	John Woods No. 4.....	do.....	do.....	1,119 L.	do.....	do.....
23	A. J. Griffith No. 1.....	Center.....	do.....	1,160 L.	do.....	do.....
24	Thomas Grove No. 1.....	do.....	Carnegie.....	1,185 B.	Gas....	do.....
25	Samuel Harvey No. 2.....	do.....	South Penn.....	do.....	do.....	do.....
26	G. P. Jams No. 1.....	do.....	N. G. W. V.....	1,280 B.	Dry....	do.....
27	Wm. Milliken No. 2.....	do.....	South Penn.....	1,130 B.	do.....	do.....
28	E. S. Scott No. 1.....	do.....	do.....	1,090 B.	do.....	do.....
29	E. T. Throckmorton No. 1.....	do.....	Carnegie.....	1,125 B.	Oil....	do.....
30	Alex. Woodruff No. 2.....	do.....	do.....	1,055 B.	Dry....	do.....
31	Biddle.....	Cumberland.....	Hukill.....	935 B.	do.....	do.....
32	Will Lynch.....	Dunkard.....	Utility.....	900	Gas....	Big Injun.....
33	Lot McClure No. 2.....	do.....	A. O. D. C.....	1,140 B.	Oil....	do.....
34	Lot McClure No. 3.....	do.....	do.....	950 B.	Gas....	do.....
35	Wm. McClure.....	do.....	Carnegie.....	1,000 B.	do.....	do.....
36	Mary A. Maple.....	do.....	Home Co.....	810 B.	do.....	do.....
37	T. J. Miller.....	do.....	Carnegie.....	1,220 B.	do.....	do.....
38	Pride No. 1.....	do.....	D. O. & G.....	1,060 B.	Oil....	do.....
39	Ira Ross.....	do.....	Carnegie.....	970 B.	Dry....	do.....
40	Gilpin South.....	do.....	do.....	880 B.	Gas....	Gantz.....
41	Stoneking No. 2.....	do.....	D. O. & G.....	1,250 B.	Oil....	Big Injun.....
42	Bell.....	Franklin.....	Carnegie.....	1,060 B.	Dry....	do.....
43	Bowlby & Co. No. 2.....	do.....	do.....	1,240 B.	Gas....	do.....
44	Bowlby & Donley No. 1.....	do.....	do.....	950 B.	do.....	do.....
45	L. M. Carpenter.....	do.....	Wheeling.....	1,020 B.	do.....	do.....
46	County poorhouse.....	do.....	Carnegie.....	960 B.	do.....	do.....
47	David Crayne No. 1.....	do.....	do.....	1,070 B.	do.....	do.....
48	Thos. Dougal No. 2.....	do.....	do.....	930 B.	do.....	Bayard.....
49	John Frye.....	do.....	West Penn.....	1,150 B.	do.....	do.....
50	Gordon heirs.....	do.....	Carnegie.....	1,280 B.	do.....	do.....
51	Rebecca Hook.....	do.....	Fort Pitt.....	950 B.	do.....	do.....
52	A. J. Lippincott No. 1.....	do.....	Carnegie.....	1,140 B.	do.....	do.....
53	John Miller.....	do.....	Fort Pitt.....	970 B.	do.....	Bayard.....
54	D. R. Pratt.....	do.....	Carnegie.....	1,250 B.	do.....	do.....
55	Wm. Rinchart No. 2.....	do.....	do.....	1,270 B.	do.....	Fifth.....
56	E. M. Sayers No. 1.....	do.....	Mfrs. L. & H.....	940 B.	do.....	Big Injun.....
57	R. A. Sayers.....	do.....	Carnegie.....	960 B.	do.....	do.....
58	Madison Scott.....	do.....	do.....	1,250 B.	do.....	Bayard.....
59	F. M. Shriver.....	do.....	do.....	1,340 B.	do.....	do.....
60	H. P. Slaughterback.....	do.....	Fort Pitt.....	970 B.	do.....	do.....
61	J. Sowers.....	do.....	do.....	935 B.	do.....	do.....
62	Strosneider.....	do.....	Carnegie.....	1,150 B.	do.....	do.....
63	Tharp No. 1.....	do.....	Fort Pitt.....	1,135 B.	do.....	do.....
64	Silas Waters.....	do.....	Carnegie.....	1,000 B.	do.....	Fifth.....
65	T. Wisecarver.....	do.....	do.....	1,075 B.	Dry....	do.....
66	J. B. Fordyce No. 1.....	Gilmore.....	Phila.....	1,098 L.	Gas....	do.....
67	T. J. L. Garrison No. 1.....	do.....	Peoples.....	1,030 B.	do.....	Big Injun.....
68	J. C. Gregg.....	Greene.....	Hukill.....	do.....	Dry....	do.....
69	L. C. Grim No. 2.....	Jackson.....	South Penn.....	1,335 B.	Oil....	Nineveh.....
70	L. C. Grim No. 5.....	do.....	do.....	1,390 B.	do.....	do.....
71	Hannah S. Grimes No. 1.....	do.....	do.....	1,225 L.	do.....	do.....
72	W. T. Grimes No. 1.....	do.....	do.....	1,478 L.	do.....	do.....
73	Thos. Hughes No. 1.....	do.....	Carnegie.....	1,085 B.	Dry....	do.....
74	Thos. Hughes No. 3.....	do.....	do.....	1,110 L.	do.....	do.....
75	Eaton Kinney No. 2.....	do.....	South Penn.....	1,280 B.	Oil....	Nineveh.....
76	Sarah M. Moore No. 1.....	do.....	do.....	1,205 L.	do.....	do.....
77	W. H. Morris No. 5.....	do.....	do.....	1,430 B.	do.....	do.....
78	J. L. Pethtel No. 2.....	do.....	South Penn.....	1,215 L.	Gas....	Gordon.....

wells in Greene County.

sea level; depths in feet.]

Pitts- burg coal.	Salt.	Big Injun.	Thirty- foot.	Gantz.	Fifty- foot.	Nine- veh.	Gor- don.	Fourth	Fifth.	Bay- ard.	Depth of well.	No. on Pl. I.
1,130	2,070	2,361			3,124	3,198					3,219	1
1,316	2,221	2,533	3,153		3,295	3,363					3,397	2
1,165	2,087	2,390			3,135	3,215					3,227	3
890	1,870	2,193	2,775		2,950	3,043	3,120	3,197			3,263	4
873	1,835	2,108	2,684	2,857	2,882	2,969	3,052	3,101				5
1,058	1,970	2,270	2,860	2,970	3,060	3,128	3,224	3,270			3,330	6
1,020		2,224			2,993	3,068	3,162	2,228			3,245	7
972					2,960	3,038	3,122	3,176			3,209	8
1,100	2,031	2,325	2,922		3,070	3,148	3,251				3,428	9
1,050	1,950	2,270	2,845			3,109	3,206				3,254	10
1,195	2,100	2,430			3,201	3,254					3,341	11
1,190	2,110	2,420	3,010		3,170	3,245					3,280	12
1,047	1,958	2,290	2,855		3,045	3,128	3,230	3,286				13
1,025	1,980	2,210			2,990	3,067					3,089	14
1,080	1,970	2,300	2,860		3,050	3,124					3,145	15
1,282	2,220	2,520			3,272	3,336					3,236	16
935	1,835	2,190			2,930	3,000					3,071	17
1,233		2,448	3,047		3,204	3,289	3,384	3,454			3,478	18
925	1,775	2,145			2,920						3,013	19
1,055	1,960	2,270	2,860		3,025	3,122	3,220		3,295	3,477		20
1,145	2,000	2,360			3,110	3,171					3,191	21
948	1,795	2,125			2,906	2,988					3,007	22
1,036	1,953	2,246			3,000	3,088					3,125	23
707	1,573	1,938	2,508		2,710	2,824	2,870	2,982	3,050			24
1,040	2,000	2,220			2,990	3,075	3,140		3,308		3,549	25
1,127	2,077	2,274	2,865		3,045	3,132	3,210	3,295	3,352		3,377	26
1,036	1,805	2,181	2,755		2,946	3,031	3,165	3,225	3,297		3,320	27
964	1,879	2,160	2,720	2,910	2,945	3,011		3,209	3,249		3,469	28
730		1,915			2,630		2,890		2,980			29
732	1,640	1,992	2,584		2,719		2,902	2,952	3,027		3,080	30
714	1,235	1,585									2,432	31
135	1,040	1,395									1,528	32
380		1,865									1,978	33
415		1,680									1,830	34
320		1,690									1,850	35
170+		1,148			1,770						1,833	36
115		1,665									1,855	37
515		1,770									1,900	38
31+		1,275		1,840	1,923		2,135			2,330	2,431	39
115		1,379		1,978							2,620	40
665		1,915									2,072	41
640		1,840			2,570		2,775	2,820	2,890	2,960	3,060	42
335		2,075			2,795		2,875	3,120	3,175	3,270	3,289	43
380		1,628			2,350		2,540	2,600	2,694	2,826	2,850	44
400	1,260	1,625	2,185	2,265	2,330	2,400	2,525	2,615	2,705	2,810	2,839	45
362	1,125	1,540		2,175	2,290				2,665	2,750	2,908	46
412		1,640		2,340	2,385		2,554	2,580	2,712		2,732	47
520		1,640			2,385		2,520	2,560	2,730	2,853	2,879	48
743	1,615	1,985	2,630		2,790		2,895	2,965	3,082	3,207	3,355	49
603		1,793		2,510	2,551		2,721	2,870		3,033	3,060	50
436	1,430	1,655	2,180		2,380		2,562	2,650	2,765	2,885	2,904	51
660	1,598		2,385	2,510	2,580		2,785	2,876	2,984	3,077	3,100	52
378	1,350		2,180				2,690	2,633	2,690	2,807	2,842	53
748		1,900			2,590		2,930		3,100	3,200	3,215	54
900		1,850		2,500	2,550				2,890		2,900	55
500	1,450	1,712	2,300	2,430			2,636		2,794	2,927	2,945	56
360		1,605			2,320		2,560	2,615	2,670	2,808	2,833	57
615		1,860		2,530	2,575		2,775		2,895	3,020	3,030	58
372		2,100		2,835	2,870		3,010	3,090	3,130	3,292	3,310	59
305	1,356	1,615	2,195				2,540	2,649	2,705	2,823	2,854	60
517	1,476	1,733			2,477		2,677	2,745	2,849	2,960	2,980	61
542		1,768			2,442		2,650	2,740	2,885	2,960		62
710	1,600	1,910			2,710		2,885		3,060	3,163	3,175	63
446		1,677		2,375	2,395		2,590	2,667	2,755	2,875	2,908	64
790		2,026		2,740	2,792			2,980	3,090	3,190	3,210	65
793	1,715	2,075	2,650			2,858	2,970	3,085	3,145			66
685	1,535	1,960									2,085	67
234	1,235	1,585									2,432	68
1,150	2,125	2,390	2,950		3,111	3,193					3,220	69
1,200	2,120	2,435	3,010		3,180	3,236					3,256	70
1,060	1,980	2,285			3,030	3,106					3,176	71
1,248	2,212	2,578	3,140		3,318	3,390					3,418	72
925		2,151				2,980					3,211	73
803		2,006		2,743	2,773		2,943	3,023	3,063		3,125	74
1,100	1,988	2,330	2,900		3,030	3,148					3,198	75
1,054	2,000	2,250			3,030	3,105					3,155	76
1,213	2,045	2,440	3,015		3,203	3,243					3,267	77
1,045	1,970	2,295			3,015	3,093	3,135				3,210	78

Summarized record of

No. on Pl. I.	Name of well.	Township.	Owner.	Eleva- tion.	Prod- uct.	Producing sand.
79	Catherine Rinehart No. 1	Jackson	South Penn.	1,043 L.	Oil	Nineveh.
80	Elisha Rinehart No. 1	do.	do.	1,110 B.	Gas	do.
81	Elisha Rinehart No. 2	do.	do.	1,470 B.	do.	Nineveh.
82	O. P. Scott No. 1	do.	Carnegie	1,065 B.	Dry	do.
83	John Eaton	Jefferson	do.	1,110 B.	Gas	do.
84	Horner No. 2	do.	Monong.	810 B.	do.	do.
85	C. M. Scott No. 1	do.	Carnegie	1,140 B.	do.	do.
86	Dr. B. Birch	Monongahela	Greenaboro.	820 B.	Dry	do.
87	A. P. Longanecker	do.	South Penn.	960	Gas	Big Injun.
88	Ben Williams	do.	Greenaboro.	960	Dry	do.
89	John Bennett	Morgan	Carnegie	1,140 B.	Gas	Bayard
90	Cotterell	do.	do.	1,070 B.	Dry	do.
91	Montgomery	do.	do.	980 B.	Gas	Bayard
92	Eliza Shape	do.	Fort Pitt	1,175 B.	Dry	do.
93	Van Kirk	do.	Jefferson	895 B.	Gas	do.
94	Hugh Auld No. 1	Morris	Nin. Pet.	1,085 B.	Dry	do.
95	Enoch Brooks No. 3	do.	South Penn.	1,455 B.	Oil	Fifty-foot
96	J. B. Carter No. 1	do.	do.	1,225 B.	do.	Nineveh.
97	James Dunn No. 1	do.	Dunn	1,090 B.	Dry	do.
98	Wm. Fonner No. 5	do.	South Penn.	1,240 B.	Oil	do.
99	Wm. Fonner No. 6	do.	do.	1,425 B.	Dry	do.
100	T. F. Lightner No. 1	do.	Carnegie	1,068 B.	do.	do.
101	Warren Mankey No. 1	do.	do.	1,255 B.	do.	do.
102	Shoup No. 5	do.	do.	1,235 B.	Oil	Gantz
103	Hugh Simpson No. 1	do.	do.	1,060 B.	Dry	do.
104	John H. Smith No. 3	do.	do.	1,070 B.	Oil	Nineveh.
105	Hester Delaney	Perry	South Penn.	970 B.	Gas	Fifth.
106	D. L. Donley	do.	Hukill	920 B.	Oil	Big Injun.
107	John Ackley No. 1	Richhill	N. G. W. V.	1,025 B.	Gas	Gordon
108	W. J. Bryan No. 1	do.	Carnegie	1,020 B.	do.	do.
109	Eva Buckingham	do.	N. G. W. V.	1,225 B.	do.	do.
110	Jas. Burns No. 1	do.	do.	1,100 B.	do.	Gordon stray.
111	Sarah Burroughs No. 1	do.	South Penn.	1,022 L.	Oil	do.
112	Durbin No. 1	do.	N. G. W. V.	1,215 B.	Gas	Big Injun.
113	Fletcher No. 1	do.	do.	1,195 B.	do.	do.
114	A. J. Headley No. 1	do.	do.	1,225 B.	Oil	Gordon
115	H. Hughes No. 1	do.	South Penn.	1,110 L.	Gas	do.
116	Jennings heirs No. 1	do.	N. G. W. V.	1,425 B.	do.	do.
117	N. H. Johnson No. 1	do.	South Penn.	1,246 L.	Oil	Nineveh
118	John Leslie No. 1	do.	do.	1,155 B.	Dry	do.
119	J. K. Loughridge No. 1	do.	Carnegie	1,255 B.	Gas	do.
120	G. W. McCullough No. 1	do.	N. G. W. V.	1,265 B.	do.	do.
121	J. D. Spragg No. 1	do.	South Penn.	1,090 B.	Dry	do.
122	Thos. Staggers No. 1	do.	do.	1,180 B.	do.	do.
123	Sugar Grove	do.	Aleppo Oil Co.	1,046 B.	Gas	do.
124	J. K. Anderson No. 1	Springhill	Carnegie	1,205 B.	do.	do.
125	Jos. Ashbee No. 1	do.	South Penn.	1,190 B.	Oil	do.
126	Silas Barnhart No. 1	do.	do.	1,555 B.	do.	Nineveh
127	Masters No. 1	do.	do.	1,075 B.	do.	Gordon
128	Cascman No. 1	do.	do.	1,255 B.	Dry	do.
129	F. A. Gover No. 2	do.	do.	1,495 B.	Oil	Nineveh
130	W. B. Griffith No. 1	do.	do.	1,365 B.	do.	Gordon
131	Hutchinson heirs No. 4	do.	do.	1,080 B.	do.	do.
132	Wm. Johnson No. 7	do.	South Penn.	1,407 L.	do.	Nineveh
133	W. H. Main No. 1	do.	do.	1,080 B.	Dry	do.
134	S. E. Martin No. 2	do.	do.	1,405 B.	Oil	Nineveh
135	W. B. Martin No. 2	do.	do.	1,399 L.	do.	do.
136	D. R. Meigham No. 1	do.	do.	do.	Gas	do.
137	Richard Fethel No. 2	do.	Peoples	do.	do.	do.
138	Wm. Petit No. 1	do.	South Penn.	1,305 B.	Oil	Nineveh
139	S. E. Phillips No. 2	do.	do.	do.	do.	do.
140	Jacob Rice No. 3	do.	do.	1,275 B.	do.	do.
141	Jacob Rice No. 14	do.	do.	1,030 L.	do.	do.
142	J. A. Riddle No. 1	do.	do.	do.	do.	Gordon
143	John Sellers No. 6	do.	do.	1,235 B.	do.	Nineveh
144	Mathias Thompson No. 1	do.	Peoples	1,195 B.	do.	do.
145	W. H. Wildman No. 6	do.	South Penn.	1,425 B.	do.	Nineveh
146	Amos Allison	Washington	Carnegie	1,100 B.	Gas	Big Injun.
147	J. W. Closser No. 1	do.	Fort Pitt	1,150 B.	do.	Fifty-foot
148	Conger	do.	do.	950 B.	do.	do.
149	Robert Hoge	do.	Carnegie	1,060 B.	Dry	do.
150	Tim Ross	do.	Ross	1,000 B.	Gas	do.
151	Geo. Stilwell	do.	Fort Pitt	1,040 B.	Dry	do.
152	Felix Bell No. 1	Wayne	do.	1,010 B.	Gas	do.
153	G. W. Blaker No. 1	do.	South Penn.	975 B.	Oil	Fifth
154	G. W. Cole No. 1	do.	do.	1,325 B.	Gas	do.
155	S. S. Eddy No. 1	do.	South Penn.	1,145 B.	Oil	do.
156	Arthur Hoy	do.	Carnegie	1,500 B.	Gas	do.

wells in Greene County—Continued.

Pitts- burg coal.	Salt.	Big Injun.	Thirty- foot.	Gantz.	Fifty- foot.	Nine- veh.	Gor- don.	Fourth	Fifth.	Bay- ard.	Depth of well.	No. on Pl. t.
890	1,730	2,110	.....	.....	2,860	2,938	.....	.....	.....	.....	2,957	79
.....	1,805	2,125	.....	.....	2,850	2,938	.....	.....	.....	.....	3,329	80
1,114	1,964	2,344	2,932	.....	3,030	3,170	.....	.....	.....	.....	3,189	81
812	.....	2,080	.....	.....	2,790	.....	2,950	.....	.....	.....	2,148	82
580	.....	1,785	.....	.....	2,550	.....	2,635	2,715	2,780	2,980	3,182	83
55	900	1,320	1,900	2,000	2,050	.....	.....	.....	.....	.....	.....	84
600	.....	1,810	.....	2,490	2,552	.....	.....	.....	.....	3,000	3,068	85
120+	.....	1,140	.....	1,705	1,800	.....	2,000	.....	.....	2,315	2,666	86
295	1,230	1,570	.....	.....	.....	.....	.....	.....	.....	.....	1,842	87
25+	926	1,316	.....	.....	.....	.....	.....	.....	.....	.....	2,120	88
580	1,480	1,800	.....	.....	2,550	.....	2,735	2,830	2,890	3,015	.....	89
465	.....	1,645	.....	2,347	2,405	.....	2,500	.....	2,730	2,855	2,906	90
523	.....	1,750	.....	2,455	2,495	.....	2,665	.....	2,800	2,956	2,975	91
776	1,670	1,975	2,555	.....	2,735	.....	2,950	3,025	3,080	3,215	3,607	92
285	.....	1,500	1,870	2,190	.....	.....	2,355	2,430	.....	.....	2,658	93
896	.....	2,060	.....	.....	.....	2,830	3,000	.....	.....	.....	.....	94
1,002	.....	2,150	.....	2,007	2,838	.....	.....	.....	.....	.....	2,964	95
1,070	2,023	2,283	.....	2,190	3,024	3,116	.....	.....	.....	.....	3,155	96
800	1,625	1,994	2,595	.....	2,725	2,855	2,940	2,985	3,070	3,200	3,265	97
902	1,914	2,100	2,700	2,308	.....	.....	.....	.....	.....	.....	2,869	98
1,025	.....	2,220	.....	2,056	2,990	.....	.....	.....	.....	.....	3,010	99
810	.....	2,035	.....	2,080	2,800	.....	2,940	2,965	.....	.....	.....	100
1,074	2,034	2,292	.....	3,105	3,031	3,125	3,215	3,292	3,350	(a)	3,651	101
897	.....	.....	.....	2,823	2,850	.....	.....	.....	.....	.....	.....	102
840	.....	1,960	.....	2,750	2,775	.....	2,990	3,030	3,115	.....	3,197	103
880	.....	2,065	2,655	.....	.....	2,935	.....	.....	.....	.....	2,970	104
505	.....	1,765	.....	.....	.....	.....	.....	.....	2,897	.....	2,927	105
368	1,260	1,635	.....	.....	.....	.....	.....	.....	.....	.....	.....	106
500	1,431	1,680	2,240	2,350	2,400	2,525	2,629	.....	.....	.....	2,640	107
482	.....	1,950	2,300	.....	2,424	.....	.....	.....	.....	.....	.....	108
620	1,560	1,790	.....	2,475	.....	.....	2,753	.....	.....	.....	.....	109
415	1,356	1,711	2,185	.....	2,325	2,445	.....	.....	.....	.....	.....	110
875	1,830	2,095	.....	.....	2,855	2,929	3,030	.....	3,110	.....	3,175	111
555	1,525	1,830	.....	.....	.....	.....	.....	.....	.....	.....	.....	112
642	1,576	1,782	2,380	2,520	2,570	2,660	.....	.....	.....	.....	2,736	113
810	1,750	2,010	2,600	.....	.....	2,854	2,935	.....	.....	.....	2,962	114
850	1,708	2,055	.....	.....	2,824	2,901	3,004	.....	.....	.....	3,133	115
780	1,717	1,940	2,570	.....	2,720	2,797	2,890	.....	2,985	.....	3,045	116
1,100	2,055	2,315	.....	.....	3,060	3,148	.....	.....	.....	.....	3,200	117
739	.....	1,910	2,530	2,630	2,690	2,768	2,902	2,935	2,990	.....	3,274	118
685	.....	1,815	.....	.....	2,600	.....	.....	.....	.....	.....	3,040	119
724	1,656	1,866	2,485	2,620	2,645	2,750	.....	2,900	.....	.....	2,978	120
890	1,805	2,105	2,665	.....	.....	.....	2,969	3,054	3,123	.....	3,227	121
1,027	1,915	2,245	.....	2,945	.....	3,073	.....	.....	.....	.....	3,334	122
614	.....	.....	.....	.....	2,595	2,662	2,825	.....	.....	.....	2,841	123
615	.....	1,820	.....	.....	2,596	.....	2,771	.....	.....	.....	3,046	124
863	1,785	2,090	2,728	.....	2,865	2,925	.....	.....	.....	.....	.....	125
1,338	2,255	2,585	.....	.....	3,318	3,380	.....	.....	.....	.....	3,398	126
890	1,838	2,130	2,700	.....	.....	3,011	3,095	.....	.....	.....	3,109	127
1,096	2,018	2,340	2,950	.....	3,130	3,195	3,272	3,305	3,390	.....	3,408	128
1,170	2,095	2,410	.....	.....	3,150	3,233	.....	.....	.....	.....	3,043	129
1,216	2,020	2,485	3,010	.....	3,190	3,260	3,383	.....	.....	.....	3,405	130
945	.....	2,189	.....	2,904	2,965	3,042	3,111	.....	.....	.....	3,200	131
1,160	2,085	2,400	.....	.....	3,040	3,203	.....	.....	.....	.....	3,226	132
782	1,725	2,038	2,591	.....	2,790	2,841	2,925	3,018	.....	.....	3,050	133
1,235	2,260	2,485	.....	.....	3,220	3,278	.....	3,452	.....	.....	3,461	134
1,105	2,030	2,345	2,920	.....	3,115	3,165	.....	.....	.....	.....	3,180	135
1,080	1,945	2,335	.....	.....	3,075	3,117	.....	.....	.....	.....	3,424	136
1,193	2,118	2,430	3,015	.....	3,176	3,243	3,369	3,430	.....	.....	3,426	137
1,000	1,925	2,230	.....	.....	2,980	3,058	.....	.....	.....	.....	3,081	138
988	1,912	2,210	.....	.....	2,968	3,037	.....	.....	.....	.....	3,057	139
1,014	1,956	2,240	2,900	.....	2,996	3,060	.....	.....	.....	.....	3,080	140
765	1,690	2,005	.....	.....	2,746	2,817	.....	.....	.....	.....	2,838	141
1,245	2,200	2,490	3,080	.....	3,230	3,313	3,408	.....	.....	.....	3,451	142
898	1,820	.....	.....	.....	2,878	2,957	.....	.....	.....	.....	2,962	143
875	1,640	2,125	2,700	.....	.....	2,957	3,040	3,094	.....	.....	3,120	144
1,182	2,100	2,420	.....	.....	3,160	3,232	.....	.....	.....	.....	2,253	145
770	.....	1,985	.....	2,700	.....	.....	.....	.....	.....	.....	3,212	146
724	1,615	1,908	2,500	2,636	2,660	.....	2,868	.....	2,999	.....	3,048	147
600	1,500	1,820	2,400	.....	2,551	.....	2,759	2,825	.....	3,057	3,165	148
655	1,565	1,855	.....	2,585	2,623	.....	2,805	2,895	2,955	.....	3,100	149
630	1,545	1,845	.....	.....	2,560	.....	2,780	2,830	2,920	3,076	3,076	150
643	1,606	1,859	.....	.....	2,640	.....	2,865	.....	2,968	3,098	3,441	151
665	.....	1,945	.....	.....	2,625	.....	2,770	2,872	2,947	.....	3,008	152
636	1,531	1,930	.....	.....	2,588	.....	.....	.....	2,932	.....	2,946	153
1,006	1,880	2,245	2,820	2,960	2,970	.....	3,270	3,290	3,345	.....	.....	154
793	1,650	2,055	.....	.....	2,740	.....	.....	.....	3,078	.....	3,093	155
1,124	2,050	2,367	2,915	3,015	3,040	.....	3,319	.....	3,450	.....	3,492	156

a Elizabeth sand, 3,580.



*Summarized record of*

No. on Pl. I.	Name of well.	Township.	Owner.	Eleva- tion.	Prod- uct.	Producing sand.
157	Thos. Hoy No. 2.....	Wayne.....	South Penn.....	1,025 B.	Oil...	Fifth.....
158	Ingham Kent No. 5.....	do.....	do.....	1,105 B.	do.....	do.....
159	Wm. Lantz No. 4.....	do.....	do.....	975 B.	do.....	do.....
160	Rachel Nichols.....	do.....	do.....	1,177 L.	Gas.....	do.....
161	Sayers & Brant.....	do.....	Carnegie.....	1,060 B.	do.....	do.....
162	H. M. Spragg.....	do.....	South Penn.....	1,050 B.	do.....	Big Injun.....
163	J. M. Stewart.....	do.....	Peoples.....	1,320 B.	do.....	do.....
164	L. L. Thomas.....	do.....	South Penn.....	1,120 B.	Oil.....	do.....
165	Abraham Tustin.....	do.....	Peoples.....	1,122 B.	Gas.....	do.....
166	Isaac Yager No. 1.....	do.....	Carnegie.....	1,075 B.	do.....	Fifth.....
167	Thos. Bayard No. 1.....	Whiteley.....	do.....	1,170	do.....	Bayard.....
168	S. J. Bradford.....	do.....	Fort Pitt.....	1,180 B.	do.....	Fifth.....
169	Thos. Mooney.....	do.....	do.....	1,110 B.	do.....	Big Injun.....
170	Zimmerman No. 1.....	do.....	Carnegie.....	1,250 B.	Gas.....	Fifth.....

## WELL RECORDS.

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*wells in Greene County—Continued.*

Pitts- burg coal.	Salt.	Big Injun.	Thirty- foot.	Gantz.	Fifty- foot.	Nine- veh.	Gor- don.	Fourth	Fifth.	Bay- ard.	Depth of well.	No. on Pl. I.
710	1,565	2,000	.....	.....	2,660	.....	.....	.....	2,990	.....	3,021	157
787	1,650	2,065	.....	.....	.....	.....	.....	.....	3,056	.....	3,079	158
628	1,506	1,900	.....	.....	2,580	.....	.....	.....	2,912	.....	2,947	159
815	1,715	2,045	2,610	.....	2,752	.....	.....	.....	3,116	.....	3,136	160
660	.....	1,970	.....	2,575	2,625	.....	2,800	2,885	2,940	.....	2,948	161
615	1,580	1,896	2,395	2,546	2,585	.....	2,839	2,913	2,931	.....	3,215	162
940	1,770	2,218	.....	.....	.....	.....	.....	.....	.....	.....	2,260	163
720	.....	2,035	.....	.....	2,640	.....	.....	.....	.....	.....	3,062	164
740	1,614	1,988	.....	.....	2,665	.....	3,027	3,056	.....	3,182	3,240	165
650	.....	1,908	.....	.....	2,570	.....	.....	.....	2,890	.....	2,910	166
555	.....	1,800	.....	2,470	2,515	.....	2,715	.....	2,835	2,960	2,965	167
698	1,632	1,936	2,470	2,615	2,640	.....	.....	.....	2,980	.....	2,995	168
645	1,560	1,890	.....	2,575	2,620	.....	2,810	.....	2,935	3,049	3,065	169
690	.....	1,920	.....	2,620	2,655	.....	2,820	2,905	2,974	.....	3,000	170

In order to facilitate reference to the wells which are numbered above 200 on the county map (Pl. I) and mentioned in the text, but whose records are not given in this report, a list giving page references is appended.

*Wells numbered above 200 which are mentioned in the text.*

No.	Name.	Page.	No.	Name.	Page.
201	Waynesburg.....	15, 64	262	H. C. Wood.....	64, 83
202	R. A. Sayers.....	16, 64, 263		Grimes.....	64
		Pl. II		Elizabeth J. Stevens.....	66
203	Benson heirs.....	44, 78, 84	265	Josephus Bowers.....	66, Pl. II
204	Lewis Kuhn heirs.....	44, 69		Iseminger No. 1.....	66
		45, 54,	267	Iseminger No. 2.....	66
205	Wm. Fonner No. 2.....	Pl. II	268	Bowen.....	67
		45, 72,	269	Evans.....	67
206	John Lewis.....	Pl. II	270	Ryan.....	67
207	Cephas Wiley.....	46	271	Gump.....	67
208	A. P. Tanner.....	49	272	Williamson.....	67
209	S. & J. L. Garard.....	50	273	Samuel Minor.....	67
210	W. L. Longanecker.....	50	274	Hoffman.....	67
211	N. H. Minor.....	50	275	Cornellison.....	68
212	G. Vance.....	49	276	Guthrie No. 1.....	68
213	Blackshire.....	51	277	John I. Worley.....	68
214	Blackshire.....	51	278	S. C. Brock No. 1.....	68
215	Stilwell.....	51	279	Tustin heirs.....	69
216	D. L. Donley.....	52	280	H. L. Granlee.....	69
217	Asa Lemley.....	52	281	T. E. Eddy.....	69
218	Williams.....	52	282	Henderson No. 2.....	69
219	J. L. Donley.....	52	283	Jacob Eddy No. 2.....	69
220	Lantz No. 1.....	57, 57	284	James Iams.....	71
221	Lantz No. 2.....	57, 57	285	Samuel Phillips.....	71
222	Lantz No. 3.....	58, 83	286	L. L. Gray.....	71
225	A. Rice.....	58, 83	287	James Carl.....	71
224	Greensboro pottery.....	58	288	James Adamson.....	72
225	Molescy.....	58	289	W. M. Boier.....	72
		58, 67,	290	A. R. White.....	72
226	Ellen Ross.....	Pl. II	291	B. L. Woodruff No. 1.....	72
227	J. Marshall.....	58, 67, 83	292	Ross heirs.....	73
228	Headley.....	58	293	A. I. Lindsey.....	73
229	Joseph Morris.....	58	294	L. C. Johnson No. 2.....	73
230	Wm. Fonner No. 1.....	54	295	Karl Crawford No. 1.....	73
231	Wm. Lantz No. 1.....	57	296	Karl Crawford No. 2.....	73
232	Rea.....	59	297	A. C. Fordyce.....	74
233	Daniel Rich.....	59	298	Elijah Maple.....	74
234	D. C. Stevenson.....	59	299	J. P. Hagan.....	74
235	Gray.....	60	300	Josephus Rice.....	74
236	Keener-Durr.....	60	301	Orndorf No. 1.....	75
237	Silas Ross.....	60	302	S. C. Leighliter.....	76
238	Mary Reed.....	60	303	Robert Ulom.....	76
239	John Steele.....	60	304	Elizabeth Sammons.....	76
240	B. F. Gabler.....	60	305	Anderson Hinerman.....	77
241	Dillner.....	62	306	Lewis Kuhn.....	77
242	Chisham.....	62	307	John Moore.....	77
243	Asa Sterling.....	62	308	John Riggie.....	77
244	Sturgiss.....	62	309	Clara Riggie.....	77
245	Jennie Miller.....	62	310	Rachel Grim.....	77
246	Stone.....	62	311	Chas. Bean.....	77
247	Hathaway.....	62	312	Thomas Thorpe.....	78
248	Hugh Keenan.....	62	313	Levi Thorpe.....	78
249	Fordyce.....	62	314	John Lewis.....	78
250	Rices Landing.....	62	315	M. L. Polen.....	78
251	Rices Landing (mill).....	62	316	Anderson.....	78
252	Degood No. 2.....	62	317	Benj. Phillips.....	78
253	Jacob Crayne.....	62	318	A. J. Dye.....	78
254	Jacob Haver.....	63	319	J. L. Riggs.....	79
255	Denny.....	63	320	Wagonroad Run.....	79
256	J. L. Luse.....	63	321	D. A. McCracken.....	83, Pl. II
257	Degood No. 1.....	63	322	S. S. Iams.....	71, Pl. II
258	Sproles.....	63	323	Michael Funk No. 3.....	Pl. II
259	Robinson.....	63	324	Brooks No. 2.....	Pl. II
260	Williams.....	63	325	Litman.....	83
261	Inghram.....	64, 83	326	John Parkinson.....	71

### TRIANGULATION STATIONS.

The exact location of Greene County with reference to latitude and longitude is determined from certain points the positions of which have been ascertained accurately by triangulation. The survey

of the county is controlled by 11 triangulation stations located within its boundaries and 9 other stations near by.

The locations of the stations within the county are shown on Plate I. by small triangles, but the accompanying sketch (fig. 7) shows the relative positions of all these points. These stations are marked by sandstone or marble posts, 6 by 6 to 12 by 12 inches in cross section and 3 or 4 feet long, set flush with the surface of the ground or projecting from 4 to 12 inches. In the center of the top of each post is cemented a bronze tablet marked "U. S. Geological Survey—Pennsylvania" or (at the West Alexander, Wilson, and Rocklick stations) "U. S. Geological Survey—West Virginia." There are two exceptions—Morley station is marked by a stone post, in the center of the top of which is cemented a copper bolt, and Oak station is a lone signal tree on the highest point of the hill.

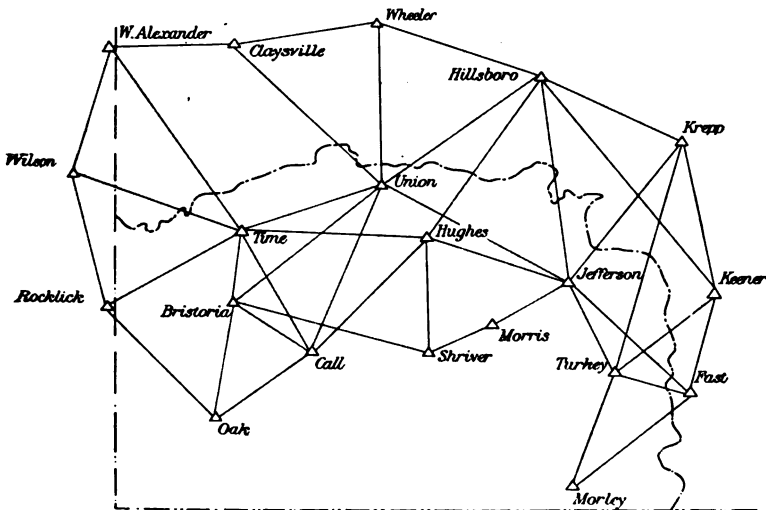


FIG. 7.—Sketch map showing location of triangulation stations on which the survey of Greene County is based.

The following descriptions are from Bulletin No. 181, with corrections:

#### BRISTORIA, GREENE COUNTY.

On a dome-shaped hill  $1\frac{1}{2}$  miles north of Bristoria and 11 miles west of Waynesburg, in a pasture owned by the Iseminger estate, about 500 feet north of a public road.

[Latitude  $39^{\circ} 53' 32.55''$ . Longitude  $80^{\circ} 23' 38.18''$ .]

To station—	Azimuth.	Back azimuth.	Log. distance.
	° ' "	° ' "	Meters.
Oak.....	6 54 26.4	186 53 51.1	4.0377279
Rocklick.....	76 18 21.5	256 13 07.0	4.0791962
Wilson.....	130 48 17.3	310 41 33.7	4.2943073
Time.....	185 30 01.5	5 30 19.2	3.8348950
Union.....	231 40 06.0	51 46 11.3	4.2357775
Call.....	301 26 01.0	121 29 33.0	3.9642161

## CALL, GREENE COUNTY.

On a cleared hill about 2½ miles southwest of Rogersville, on land owned by Robert Call.

[Latitude 39° 50' 56.68". Longitude 80° 18' 07.61".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Oak.....	56	45	12.3	236	41	05.3	4.0403119
Bristoria.....	121	29	33.0	301	26	01.1	3.9642757
Time.....	148	14	24.8	328	11	10.5	4.1355279
Union.....	200	05	07.7	20	07	40.7	4.2167757
Hughes.....	221	22	02.6	41	26	18.6	4.1564204
Shriver.....	268	59	33.8	89	04	36.7	4.0507482

## CLAYSVILLE, WASHINGTON COUNTY.

On a cone-shaped hill 1 mile south of Claysville, on cultivated land belonging to J. Mosier.

Reference marks: Double chestnut tree, south, 72 feet; apple tree, east, 80 feet.

[Latitude 40° 06' 20.01". Longitude 80° 23' 55.46".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Wheeler.....	257	55	51.0	78	01	54.2	4.1350905

## FAST, FAYETTE COUNTY.

About 2 miles southeast of Masontown, on road to Smithfield, 8 feet north of an east-west fence, on land owned by Mr. Fast, who lives about 300 yards south of station.

[Latitude 39° 49' 29.36". Longitude 79° 53' 30.97".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Morley.....	47	17	02.2	227	11	58.5	4.1871165
Turkey.....	99	01	56.6	278	58	14.5	3.9215699
Jefferson.....	129	24	42.4	309	19	08.7	4.2041975
Keener.....	190	44	15.0	10	45	03.1	3.9804006

## HILLSBORO, WASHINGTON COUNTY.

In a rocky pasture, about 1,000 feet north of Scenery Hill cross-roads, along the National Pike, 3 miles northwest of Beallsville, on land owned by Mrs. E. S. Tonebaugh.

[Latitude 40° 05' 17.67". Longitude 80° 04' 15.81".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Krepp.....	295	07	06.8	113	13	26.6	4.1890430
Jefferson.....	351	12	54.0	171	14	13.9	4.2860956
Keener.....	319	14	33.3	139	22	16.0	4.4181154
Hughes.....	33	02	20.9	212	51	42.6	4.2751190
Union.....	51	48	48.2	231	42	26.5	4.2531028
Wheeler.....	108	08	43.9	288	02	07.1	4.1859700

# TRIANGULATION STATIONS.

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## HUGHES, GREENE COUNTY.

On a hill covered with an orchard of small peach trees, about 3½ miles north of Waynesburg, owned by the Hughes estate.

[Latitude 39° 56' 45.33". Longitude 80° 11' 26.53".]

To station—	Azimuth.	Back azimuth.	Log. distance.
	° ' "	° ' "	Meters.
Call.....	41 26 18.6	221 22 02.6	4.1564204
Union.....	141 03 49.6	321 02 06.5	3.7827879
Hillsboro.....	212 57 42.6	33 02 20.9	4.2751190
Jefferson.....	283 56 38.3	104 02 35.8	4.1344974
Morris.....	322 34 23.6	142 37 23.6	4.0403635
Shriver.....	350 35 28.6	170 36 15.8	4.0296854

## JEFFERSON, GREENE COUNTY.

About 1½ miles southeast of Jefferson, on a high, bald knob owned by Lawrence Kraft.

[Latitude 39° 54' 58.46". Longitude 80° 02' 11.55".]

To station—	Azimuth.	Back azimuth.	Log. distance.
	° ' "	° ' "	Meters.
Hillsboro.....	171 14 13.9	351 12 54.0	4.2860956
Krepp.....	221 23 32.6	41 28 31.9	4.2228713
Keener.....	273 01 04.2	93 07 20.3	4.1512351
Fast.....	309 19 08.7	129 24 42.4	4.2041975
Turkey.....	334 56 40.0	154 58 31.5	3.9895121
Hughes.....	104 02 35.8	283 56 38.3	4.1344974
Union.....	115 15 13.2	295 07 32.4	4.2747311

## KEENER, FAYETTE COUNTY.

About 6 miles east of Carmichaels, 1½ miles north of McClellandtown, and 23.3 feet west of a lone locust tree on a bare knob owned by Ben Keener, who lives 300 yards south of station.

Reference mark: A stone post, 36 by 12 by 12 inches, set 34 inches in the ground, in the center of top of which is cemented an aluminum bolt; azimuth from station, 275° 27'; distance, 20 feet.

[Latitude 39° 54' 33.86". Longitude 79° 52' 16.00".]

To station—	Azimuth.	Back azimuth.	Log. distance.
	° ' "	° ' "	Meters.
Fast.....	10 45 03.1	190 44 15.0	3.9804006
Turkey.....	51 08 33.2	231 04 02.8	4.1097964
Jefferson.....	93 07 26.3	273 01 04.2	4.1512351
Hillsboro.....	139 22 16.0	319 14 33.3	4.4181154
Krepp.....	166 57 58.6	346 56 35.3	4.1346730

## KREPP, WASHINGTON COUNTY.

About 1½ miles northwest of Brownsville, on a prominent and well-known bald knob owned by James Nickson.

[Latitude 40° 01' 44.55". Longitude 79° 54' 25.69".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Jefferson.....	41	28	31.9	221	23	32.6	4.2228714
Hillsboro.....	115	13	26.6	295	07	06.8	4.1590430
Keener.....	346	56	35.3	166	57	58.6	4.1346730
Turkey.....	18	00	10.1	197	57	02.7	4.3515150

## MORLEY, GREENE COUNTY.

On a flat, bald ridge owned by D. W. Morley, 1 mile southwest of Bald Hill and one-half mile north of the Pennsylvania-West Virginia State line. There are a few trees under the brow of the hill on the east side.

[Latitude 39° 43' 50.65". Longitude 80° 01' 25.65".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Turkey.....	194	30	55.5	14	32	17.4	4.0842057
East.....	227	11	58.5	47	17	02.2	4.1871165

## MORRIS, GREENE COUNTY.

On land owned by Henry Morris, 5 miles southeast of Waynesburg, at the head of Braden Run in a cleared field 600 feet north of the ridge road and one-fourth mile west of Mr. Morris's house.

[Latitude 39° 52' 2.67". Longitude 80° 6' 47.91".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Shriver.....	09	27	10.8	249	24	58.1	3.7205630
Hughes.....	142	37	23.6	322	34	23.6	4.0403635
Jefferson.....	230	25	41.6	50	28	38.8	3.9301943

## OAK, GREENE COUNTY.

On a high, cleared hill 1½ miles west of Nettle Hill, 3 miles south of Higbee and 5½ miles south of Bristoria, on land owned by S. T. Williams.

[Latitude 39° 47' 41.45". Longitude 80° 24' 33.31".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Rocklick.....	127	39	19.7	307	34	40.8	4.1163332
Bristoria.....	186	53	51.1	6	54	26.4	4.0377279
Call.....	236	41	05.3	56	45	12.3	4.0403119

# TRIANGULATION STATIONS.

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## ROCKLICK, MARSHALL COUNTY, W. VA.

On a bare hill one-half mile south of the town of Rocklick, W. Va.  
A county road passes on the north side of the hill.

[Latitude 39° 52' 00.15". Longitude 80° 31' 48.73".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Wilson.....	168	13	33.5	348	12	04.9	4.2053434
Time.....	231	50	19.0	51	55	51.4	4.1943297
Bristoria.....	256	13	07.0	76	18	21.5	4.0791962
Oak.....	307	34	40.8	127	39	19.7	4.1163332

## SHRIVER, GREENE COUNTY.

On a bald hill 2 miles west of Randolph Church and 3 miles south of Waynesburg, on land owned by William Shriver.

[Latitude 39° 51' 02.82". Longitude 80° 108' 14.90".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Call.....	89	04	36.7	268	59	33.8	4.0507482
Hughes.....	170	36	15.8	350	35	28.6	4.0290854
Morris.....	249	24	58.1	69	27	10.8	3.7205630

## TIME, GREENE COUNTY.

In a pasture owned by the Miller estate on a cleared knob 2½ miles north of Graysville and 1 mile west of Time post-office.

[Latitude 39° 57' 13.23". Longitude 80° 23' 10.56".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Bristoria.....	5	30	19.2	185	30	01.5	3.8348950
Rocklick.....	51	55	51.4	231	50	19.0	4.1943297
Wilson.....	111	17	45.4	291	10	43.8	4.2228433
Union.....	253	14	48.2	73	20	36.0	4.1275611
Call.....	328	11	10.5	148	14	24.8	4.1355279

## TURKEY, GREENE COUNTY.

About 1½ miles west of Sigsbee and 4 miles south of Carmichaels, on Turkey Knob, in a cultivated field owned by Leroy Hartley.

[Latitude 39° 50' 11.72". Longitude 79° 59' 17.66".]

To station -	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Morley.....	14	32	17.4	194	30	55.5	4.0842057
Jefferson.....	154	58	31.5	334	56	40.0	3.9895121
Krepp.....	197	57	02.7	18	00	10.1	4.3515150
Keener.....	231	04	02.8	51	08	33.2	4.1097964
Past.....	278	58	14.5	99	01	56.6	3.9215699



## UNION, GREENE COUNTY.

In a pasture on a prominent hill 7 miles north-northwest of Waynesburg and 2½ miles east of Deerlick.

[Latitude 39° 59' 18.24". Longitude 80° 14' 09.16".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Call.....	20	07	40.7	200	05	0.7	4.2167757
Bristoria.....	51	46	11.3	231	40	06.0	4.2357775
Time.....	73	20	36.0	253	14	48.2	4.1275671
Wheeler.....	178	04	16.4	358	04	01.9	4.2002916
Hillsboro.....	231	42	26.5	51	48	48.2	4.2531028
Jefferson.....	295	07	32.4	115	15	13.2	4.2747311
Hughes.....	321	02	06.5	141	03	49.6	3.7827879

## WEST ALEXANDER, OHIO COUNTY, W. VA.

On a hill 1 mile southwest of West Alexander, Pa.

Reference marks: A maple tree 2 feet in diameter bears N. 85° 56' W., distance, 72.1 feet; a butternut tree 10 inches in diameter bears S. 52° 25' E., distance, 15 feet.

[Latitude 40° 03' 48.62". Longitude 80° 31' 41.59".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Wilson.....	19	15	19.31	199	13	46.06	4.0179782

## WHEELER, WASHINGTON COUNTY.

On a sparsely timbered hill, 3 miles south of Washington, on land of William Courson, who lives at the north base of the hill.

[Latitude 40° 07' 52.16". Longitude 80° 14' 31.71".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Claysville.....	78	01	54.2	257	55	51.0	4.1350995
Hillsboro.....	288	02	07.1	108	08	43.9	4.1859700
Union.....	358	04	01.9	178	04	16.4	4.2002916

## WILSON, MARSHALL COUNTY, W. VA.

On the farm of G. H. Wilson, 1½ miles northeast of Sand Hill and 2½ miles west of West Union, near the line fence between the lands of Mr. Wilson and Mr. Howard, where the fence crosses the summit of the hill. The Moundsville and Washington pike passes over the north side of the hill.

[Latitude 40° 00' 29.42". Longitude 80° 34' 06.77".]

To station—	Azimuth.			Back azimuth.			Log. distance.
	°	'	"	°	'	"	Meters.
Time.....	291	10	43.8	111	17	45.4	4.2228433
Bristoria.....	310	41	33.7	130	48	17.3	4.2943973
Rocklick.....	348	12	04.9	168	13	33.5	4.2053434

## BENCH MARKS.

The elevations in the following list are based on and adjusted between bench marks established by spirit leveling. All bench marks are referred to an aluminum tablet in the foundation of the Seventh Avenue Hotel at Pittsburg, marked "738 Pittsburg 1899," the elevation of which is accepted as 738.384 feet above mean sea level.

The leveling was done as follows: On the Waynesburg quadrangle in 1900, under the direction of Frank Sutton, topographer, by J. H. Wetzel, levelman; on the Blacksville quadrangle in 1901, under A. H. Bumstead, topographer, by M. P. Page, levelman; on the Rogersville quadrangle, mostly in 1902, under R. D. Cummin, topographer, by Charles Hartman, jr., levelman; and on the Amity quadrangle, partly in 1901, under A. H. Bumstead, topographer, by Messrs. Page and H. Wood, levelmen; partly in 1902, under R. D. Cummin, topographer, by Charles Hartman, jr., levelman; and partly in 1903, under Mr. Sutton, by Mr. Wetzel. The work on the Claysville quadrangle, was done in 1905, under W. T. Griswold, topographer, by B. J. Green, levelman.

The standard bench marks except that at Mount Morris are stamped "PITTS" or "PITTSBURG" in addition to figures of elevation, which, being taken from unadjusted field values, are in many cases incorrect. For instance, the tablet at Waynesburg court-house is marked "1035" but its actual elevation is 1,034.446 feet above tide. The localities here described are arranged alphabetically by townships.

## ALEPPO TOWNSHIP.

	Feet.
Aleppo post-office, or Bridgeport, highway bridge over South Fork Dunkard Fork Wheeling Creek, in southwest abutment; bronze tablet marked "1060 PITTS".....	1, 059. 588
Aleppo, 1.1 miles south of, at forks of roads near Sam King's residence, on ledge of rock; chiseled square.....	1, 134. 05
Aleppo 2.3 miles southwest of, near top of ridge, at forks of road to Sugar Grove, Aleppo, and Waynesburg, on ledge of rock; chiseled square.....	1, 462. 34
McCracken, 1 mile east of, near forks of roads to Jacksonville and Ryerson station, near sawmill, at entrance to John Lewis's residence, on stone step: chiseled square.....	999. 29
Morford post-office, 1 mile northwest of, between Antill's residence and post-office, on rock near hickory tree; chiseled square.....	1, 500. 34
Morford post-office, Jacob McMiller's residence next to, on stone step at entrance; chiseled square.....	1, 394. 88

## CENTER TOWNSHIP.

Bluff, 0.2 mile north of, in front of A. R. White's residence at bottom of hill, on stone; chiseled square.....	1, 178. 81
Bluff, 1.5 miles north of, near corner of fence, in front of schoolhouse, at forks of road, on stone; chiseled square.....	1, 080. 88

	Feet.
Hunters Cave, at center of crossroads to Nineveh, Waynesburg, Rogersville, and Graysville; cut in large ledge of rock .....	1, 334. 02
Hunters Cave, 0.5 mile north of, at bottom of hill, barn near E. Cutter's residence, on first step of foundation wall; square cut .....	1, 144. 47
Rogersville, highway bridge over Tenmile Creek at, southwest abutment, set in capstone; aluminum tablet marked "978 PITTS" .....	977. 337
Rogersville, 1 mile north of, on ledge of rock; chiseled square .....	1, 051. 76
Rogersville, 2.5 miles north of, at top of hill, residence of Hiram Ely, on stone step; cut .....	1, 393. 86
Rogersville, 1.5 miles east of, on Waynesburg road, bridge over small stream, on stone; chiseled square .....	965. 97
Rogersville, 3 miles east of, opposite R. H. Katz's residence, on Waynesburg road, on ledge of rock; chiseled square .....	968. 47
Rogersville, 1 mile south of, covered bridge over Hargus Creek, on southwest abutment; chiseled square .....	982. 21
Rogersville, 2 miles south of, near schoolhouse, covered bridge over Hargus Creek, on southwest abutment; chiseled square .....	1, 007. 70
Rogersville, 3 miles south of, at end of small bridge near Inghram residence, on rock; chiseled square .....	1, 038. 03
Rogersville, 4.5 miles south of, at entrance to church, on stone; chiseled square ..	1, 053. 75

## CUMBERLAND TOWNSHIP.

Carmichaels, on stone step of entrance to schoolhouse; aluminum tablet marked "1004 PITTSBURG" .....	1, 004. 297
Carmichaels, 2 miles west of, covered bridge over Muddy Creek, on southwest wall of; chisel mark .....	981. 76
Carmichaels, 2.5 miles west of, opposite brick and white house, on stone water trough, chisel mark .....	1, 004. 94
Khedive, residence of Mrs. Sarah Yoder, in west end of, on foundation stone under bay window; aluminum tablet marked "1013 PITTSBURG" .....	1, 013. 255

## DUNKARD TOWNSHIP.

Davistown, middle (wooden) bridge in, in ledge of rocks at end of northeast wing wall; bronze tablet marked "932 PITTSBURG" .....	932. 174
Davistown, 1.5 miles southeast of, iron bridge over Dunkard Creek, or northwest wing wall; chisel mark .....	871. 78
Dilliner, 1 mile north of, bridge over Dunkard Creek, at junction with Monongahela River, west side of south abutment; stone, chisel marked .....	799. 927
Dunkard, 0.5 mile west of, bridge on northwest wing wall; chisel mark .....	848. 8

## FRANKLIN TOWNSHIP.

Waynesburg, court-house, northeast corner of stone veranda; bronze tablet marked "1035 PITTSBURG" .....	1, 034. 446
Waynesburg, east end of, covered bridge over Tenmile Creek, on southeast wing wall; chisel mark .....	934. 89
Waynesburg, 2 miles south of, on rock at bridge; chisel mark .....	968. 79
Waynesburg, 5 miles south of, floor of iron bridge; chisel mark .....	1, 037
Waynesburg, 1 mile west of, junction of road to Oak Forest and Sycamore; small spike in telegraph pole .....	939. 78
Waynesburg, 2 miles west of, on Rogersville road, on ledge of rock; chiseled square .....	956. 77
Waynesburg, 3 miles west of, highway bridge on Rogersville road, on southeast abutment; chiseled square .....	952. 78

## BENCH MARKS.

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## GILMORE TOWNSHIP.

	Feet.
Ashtree, on stone in front of R. Gray's residence; chiseled square.....	1, 320. 19
New Freeport, 2.5 miles east of, 100 feet east of William Phillips's residence, at bend in road, on ledge of rock; chiseled square.....	1, 191. 81
Pinebank, 0.5 mile west of, public bridge over Blockhouse Run, on northwest abutment; chiseled square.....	1, 012. 97
Pinebank, highway bridge over Toms Run at, in southeast abutment; bronze tablet marked "1006 PITTS".....	1, 005. 454

## JACKSON TOWNSHIP.

Bluff, 0.5 mile southwest of, on north side of road near bottom of hill, on ledge of rock; chiseled square.....	1, 222. 28
Pinebank, 2.1 miles north of, at forks of road leading to Bluff and Nettle Hill, on ledge of rock; chiseled square.....	1, 079. 14
Triumph, 0.8 mile west of, residence of Lindly Roberts, on flat stone at entrance; chiseled square.....	1, 083. 63

## JEFFERSON TOWNSHIP.

Clarksville, 1 mile south of, covered bridge over Tenmile Creek, northwest wing wall, on coping stone (next to bridge); bronze tablet marked "814 PITTS-BURG".....	814. 167
Jefferson, 0.5 mile north of, covered bridge, on southwest wing wall; chisel mark.....	851. 24

## MORRIS TOWNSHIP.

Nineveh, entrance to Van Cleve Hotel, in southeast top of foundation wall; aluminum tablet marked "1023 PITTS".....	1, 022. 180
Nineveh, 0.8 mile north of, near Smith's residence, highway bridge over Browns Fork, on northeast abutment; chiseled square.....	1, 048. 01
Nineveh, 2 miles northwest of, near Caldwell's residence, near barn, on ledge of rock; chiseled square.....	1, 168. 19
Nineveh, 2.5 miles west of, on top of hill at crossroads to Washington, Waynesburg, Simpson Store, and Nineveh; stone at cut.....	1, 447. 58
Nineveh, 3 miles west of, at bottom of hill, north side, on ledge of rock; chiseled square.....	1, 251. 38
Nineveh, 0.5 mile southeast of, 300 feet northwest of greenhouse, on ledge of rock; chiseled square.....	1, 019. 73
Nineveh, 2 miles southeast of, at Y roads, 6 miles to Rutan, on ledge of rock; chiseled square.....	988. 60
Time, 0.2 mile east of, at forks of road, on ledge of rock; chiseled square.....	1, 153. 54
Time, 0.7 mile northwest of Ewing's residence, on large boulder used as embankment for small run in front of; chiseled square.....	1, 044. 38

## PERRY TOWNSHIP.

Mount Morris, 150 feet west of mill, in northwest abutment of bridge over Robinson Run, 8 feet from north end of bridge, 10 feet from center of road; bronze tablet stamped "904 GRAFTON".....	903. 840
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## RICHHILL TOWNSHIP.

Bradens Mill, 1.6 miles northwest of Video, highway bridge over Big Wheeling Creek, in capstone of southeast abutment; bronze tablet marked "918 PITTS".....	917. 211
Crabapple, at forks of road, highway bridge over Crabapple Creek, on abutment; chiseled square.....	954. 85

	Feet.
Crabapple, 1 mile northeast of, near F. M. Braddock's residence, highway bridge over Crabapple Creek, on abutment; chiseled square.....	986. 10
Durbin, iron bridge over Crabapple Creek, on abutment; chiseled square.....	896. 42
Durbin, 1.2 miles south of, at forks of road, near S. Lyon's residence, highway bridge over Wheeling Creek, on abutment; chiseled square.....	922. 72
Eno, on top step of entrance to church at; chiseled square.....	997. 59
Eno, 1 mile west of, on north side of road along Wheeling Creek, on ledge of rock; chiseled square.....	992. 62
Ryerson Station, Dunkard Fork of Wheeling Creek at, highway bridge over, on abutment; chiseled square.....	931. 29
Ryerson Station, 1.6 miles south of, at forks of roads leading to Cameron, Ryerson, and Aleppo, and near Bryan's residence, on stone; chiseled square...	958. 61
Ryerson Station, 3 miles south of, near Earnest's residence, covered bridge over Dunkard Fork of Wheeling Creek, on abutment; chiseled square.....	974. 93
Simpson Store, 0.5 mile west of; at spring, on ledge of rock; chiseled square...	1, 013. 97
Simpson Store, 3 miles west of, iron bridge over Big Wheeling Creek, on northwest abutment; chisel mark.....	963. 20
Simpson Store, 3.5 miles west of, bridge over Big Wheeling Creek, on northwest abutment; square cut.....	944. 38
Video, 0.5 mile west of, on top of ridge, entrance to Frank D. Headley's residence, on corner of flagging; chiseled square.....	1, 346. 99

## SPRINGHILL TOWNSHIP.

Deep Valley, public bridge over Fish Creek at, on northeast abutment; square cut.....	1, 015. 81
Deep Valley, 0.5 mile west of, covered bridge over Wagonroad Run, on southwest abutment; chiseled square.....	1, 004. 67
Deep Valley, 2 miles west of, 2 miles east of State line, on north side of road near house, on rock under walnut tree; chiseled square.....	996. 20
Morford post-office, 0.6 mile south of, in roadway near entrance to John Miller's residence, on ledge of rock; chiseled square.....	1, 466. 46
Morford post-office, 2 miles southeast of, at forks of roads on Wagonroad Run, near residence of John Calwell, on face of rock at bottom of hill; chiseled square.....	1, 094. 68
New Freeport, public bridge over Fish Creek at, in southwest abutment; bronze tablet marked "1062 PITTS".....	1, 062. 006
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New Freeport, 0.5 mile west of, bridge over Herod Run, on top of northeast abutment; paint mark.....	1, 048. 53
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## WASHINGTON TOWNSHIP.

Ruff Creek, wooden dwelling house of John Breston, northeast corner, in upper foundation stone; bronze tablet marked "999 PITTSBURG".....	997. 697
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## WHITELEY TOWNSHIP.

Newton, on northeast wing wall of iron bridge, 2 coping stones from bridge; bronze tablet marked "1010 PITTSBURG".....	1, 009. 525
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[Bulletin No. 304.]

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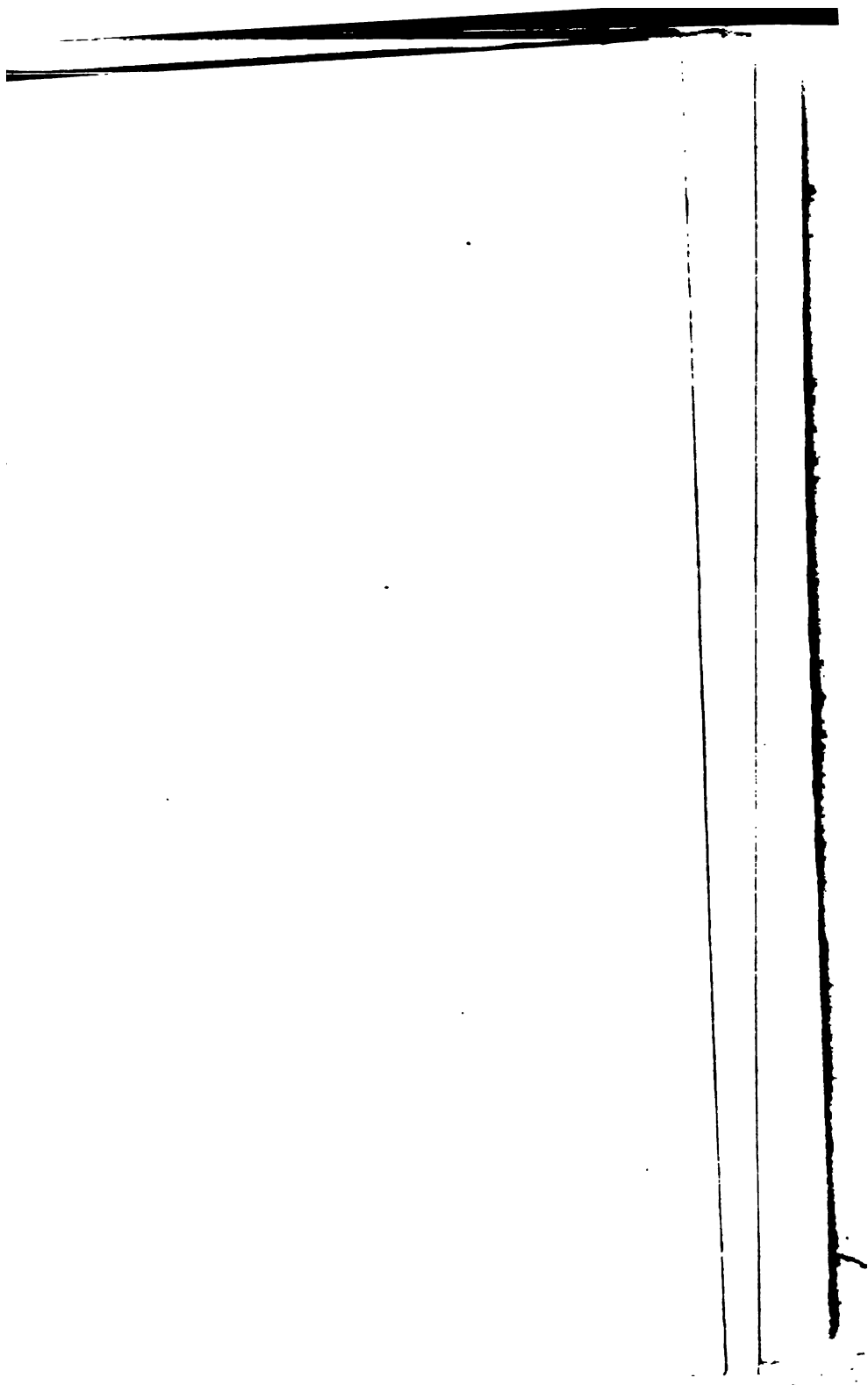
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1. The first part of the document is a list of the names of the persons who were present at the meeting.

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# THE ANALYSIS OF SILICATE AND CARBONATE ROCKS

BY  
W. F. HILLEBRAND



WASHINGTON  
GOVERNMENT PRINTING OFFICE  
1907

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# THE ANALYSIS OF SILICATE AND CARBONATE ROCKS.

By W. F. HILLEBRAND.

## INTRODUCTION.

### OBJECT AND SCOPE OF THE PRESENT TREATISE.

The literature relating to analysis of silicates is extensive but scattered, and in no single paper up to the time of the publication of Bulletin No. 148 of this Survey was there to be found in the light of modern methods, a satisfactory exposition of the procedures to be followed or the precautions to be observed, especially in the search for some of the rarer constituents or those which, without being rare, have been of late years recognized as occurring persistently in small amounts. It was not the intention to make the chapter on rock analysis in that bulletin a manual on mineral or even rock analysis, but to show primarily the principles and methods by which the major part of the analyses made up to that time in the laboratory of the Geological Survey had been conducted, and thus to afford a partial measure of the trustworthiness of those analyses. At the same time it was felt that the experience gained by the chemists of the Survey since the establishment of its first chemical laboratory in Denver might be useful to most chemists engaged in mineral and rock analysis. The favorable reception accorded it by chemists led to the republication of a portion of Bulletin No. 148 in more extended form as Bulletin No. 176. Since the publication of the latter bulletin in 1900 two other treatises on rock analysis have appeared: Manual of the Chemical Analysis of Rocks, by Dr. H. S. Washington (1904), and Anleitung zur Gesteinsanalyse, by Dr. Max Dittrich (1905). The former is based largely on Bulletin No. 176; the latter, while adopting some of the methods advocated in that bulletin, adheres more to methods which are in greater favor in Germany than in this country. They both differ from Bulletin No. 176 in devoting less space to a discussion of principles and to the determination of the less common constituents of rocks. Doctor Washington's book enters into considerable detail in describing manipulations, since it was written



for chemists, petrologists, mining engineers, and others who have not made a particular study of quantitative analysis. These books have not, therefore, rendered the Survey publication superfluous. On the contrary, since some methods have undergone improvement or been superseded by better ones, the time seems ripe for its republication in revised form.

Experience has shown that for the use of the student the outlines of some methods and procedures were too briefly sketched in the earlier publications. Where it seemed called for, therefore, these have received considerable elaboration.

For the reasons that the chief carbonate rocks form so important an element in the composition of the earth's crust, and that the knowledge of their composition is of moment to the geologist as well as to the cement maker, who now makes enormous use of them, a special section devoted to their analysis has been added. The methods applied to their analysis differ in but unimportant respects from those used with the more siliceous rocks, since they are to a great extent themselves siliceous, contain essentially the same constituents, and therefore the same principles apply to both. Naturally they demand and receive briefer treatment.

The special problems often arising in the analysis of rocks of extra-terrestrial origin—the more or less stony meteorites—will not be considered here. An analysis of that kind should never be intrusted to the novice, but only to the chemist who has a knowledge of the composition and properties of the peculiar mineral constituents of those bodies and a judgment fit to cope with the oftentimes difficult problems presented by them.

The methods described in the following pages are not restricted altogether to those actually in use in the Survey laboratory. A number of alternative ones are given which are known or believed to be good, in order that those who may wish to use this treatise as a practical guide shall have some choice of selection in case the expensive apparatus or complicated arrangements sometimes preferred are not available. Where silicate and limestone analyses are very frequently made, however, it is a saving of time and money in the end to set up permanent arrangements for convenience in determining water, carbon dioxide, ferrous iron, in making reductions in hydrogen, etc.

It was my intention to prepare a chapter on the mechanical separation in a small way of minerals by the various processes more or less in vogue, as panning, upward flow of water, heavy solutions, and magnetism, but lack of time, and in fact insufficient experience with some of the methods and solutions, have prevented its development. To those seeking information on some of these methods reference is made to the works of Rosenbusch-Wülfing, *Mikroskopische Physiographie der Mineralien und Gesteine*, volume 1 (1904); Rosenbusch-

Iddings, *Microscopical Physiography of Rock-making Minerals*, fourth edition (1903); and Ferdinand Zirkel, *Lehrbuch der Petrographie*, second edition, volume 1.

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# SILICATE ROCK ANALYSIS.

## PART I.—INTRODUCTION.

### 1. IMPORTANCE OF COMPLETE AND THOROUGH ANALYSES.

The composition of the ultimate ingredients of the earth's crust—the different mineral species which are there found and of many of which its rocks are made up—was the favorite theme of the great workers in chemistry of the earlier half of the nineteenth century, and for the painstaking care and accuracy of Berzelius, Wöhler, and others the mineralogists and geologists of to-day have reason to be thankful. Considering the limited facilities at their disposal in the way of laboratory equipment and quality of reagents, the general excellence of their work is little short of marvelous. As an outgrowth of and closely associated with the analysis of minerals came that of the more or less complex mixtures of them—the rocks—to aid whose study by the petrographer and geologist a host of chemists have for many decades annually turned out hundreds of analyses of all grades of quality and completeness. With the growth and extraordinary development of the so-called organic chemistry inorganic chemistry gradually fell into a sort of disfavor. In many, even the best, European laboratories the course in mineral analysis, while maintained as a part of the curriculum of study, became but a prelude to the ever expanding study of the carbon compounds, which, multiplying rapidly and offering an easy and convenient field for original research and possible profit, proved more tempting to young chemists than the often worked over and apparently exhausted inorganic field. For one student devoting his time to higher research on inorganic lines fifty perhaps were engaged in erecting the present enormous structure of carbon chemistry. The instruction afforded the student in mineral analysis was confined to the ordinary separations of the commoner ingredients occurring in appreciable quantities, with little regard to supposed traces and with still less attempt to find out if the tabulated list really comprised all that the mineral or rock contained.

With the introduction of improved methods of examination by the petrographer, especially as applied to thin rock sections, and the use

of heavy solutions, whereby, on the one hand, the qualitative mineral composition of a rock could be preliminarily ascertained with considerable certainty, and on the other chemical examination of the more or less perfectly separated ingredients was rendered possible, a great help and incentive was afforded to the few chemists engaged in rock analysis. The microscope often obviated in part the necessity for tedious and time-wasting qualitative tests, and the heavy solutions, by permitting the concentration and separation of certain components, facilitated the detection of elements whose existence had long been overlooked.

Meanwhile, in the progress of chemistry new methods and reagents for qualitative detection and quantitative separation and estimation were gradually being devised and discovered. The belief that some well-established methods were adequate was shown to be unwarranted; some had to be discarded altogether; others were still utilizable after modification. In the light thus shed it became possible to explain many hitherto incomprehensible variations in the composition of some rock species or types, as shown in earlier analyses, and in not a few cases it appeared that the failure to report the presence of one or more elements had obscured relations and differences which more thorough examination showed to exist (see p. 17). Consequently there arose a feeling of distrust of much of the older work in the minds of those chemists and petrographers who were best fitted to judge of its probable qualities. This, and the incompleteness of nearly all the earlier work (and much of that of to-day, unfortunately), as shown by the largely increased list of those elements now known to enter into the normal composition of rocks, are rendering the old material less and less available to meet the increasing demands of the petrographer.<sup>a</sup>

And yet these demands on his part are, with few exceptions, by no means so exacting as they should be. Frequently the analysis is intrusted to a student without other experience than that gained by the analysis of two or three artificial salts and as many comparatively simple natural minerals, and with a laboratory instructor as adviser whose experience in rock analysis may be little superior to his own. In other words, one of the most difficult tasks in practical analysis is expected to be solved by a tyro, and his results are complacently accepted and published broadcast without question. Even to those thoroughly familiar with the subject rock analysis is a complex and often trying problem. Although long practice may have enabled

<sup>a</sup> Dr. Henry S. Washington, in Professional Papers Nos. 14 (Chemical analyses of igneous rocks published from 1884 to 1900, with a critical discussion of the character and use of analyses) and 28 (The superior analyses of igneous rocks from Roth's Tabellen, 1869 to 1884, arranged according to the quantitative system of classification) of the United States Geological Survey, has done a most important work in fitting the great mass of data accumulated in the thousands of analyses published since 1869. After a systematic critical examination he has assigned to each analysis a certain value on a scale of 5. Many of these values may be incorrect, but in most cases they undoubtedly give a fairly true measure of the weight to be attached to a particular analysis.

one to do certain parts of it almost mechanically, perplexing questions still arise which require trained judgment to properly meet and answer, and there is yet room for important work in some of the supposedly simplest quantitative determinations. If the results are to have any decided value for purposes of scientific interpretation and comparison, they must be the outcome of the work of one who is able to find his way through the intricacies of an analysis in which from fifty to twenty-five components are to be separated and estimated with close approach to accuracy, and this a beginner can not hope to do. A conscientious chemist should have a live interest in this matter. He should work with a twofold purpose in view—that of lightening the labors of those who come after him by enabling them to use his work with less supplementary examination, and that of enhancing his own reputation by meriting encomiums on work that has stood the test of time.

How little understood may be the principles underlying the treatment of bodies so complex and the accurate separation and determination of their constituents, even when these are comparatively few in number, has been strikingly shown during the last five years in the work of several committees of chemists charged with the investigation of the methods employed in various lines of technical chemistry involving the analysis or assay of zinc ores, slags from the smelting of copper ores, argillaceous limestones, and cements. In all cases a marked inability to obtain agreeing results is apparent, not only among those less experienced, but among those supposed to be more expert in each of the particular fields as well. Some improvement in silicate analysis has resulted from the investigations set on foot and the recommendations made by these committees, and further improvement may be expected, but the situation is yet anything but satisfactory. The art of analysis stands in great need of more thorough treatment in our educational institutions.

The petrographer, again, should seek to have his analyses made as complete as possible, and not, as is so often the case, be content with determinations of silica, alumina, the oxides of iron, lime, magnesia, the alkalies, and water. The latter course, it is true, is entirely justifiable at times, and may serve the immediate purposes of the analyses, but their incompleteness may, on the other hand, not only conceal points fruitful of suggestion to the attentive mind, but, what is of still greater importance, they may be actually misleading. Numerous instances of totally inaccurate conclusions to be drawn from them have fallen under my own observation to fully justify this plea in favor of greater completeness in rock and mineral analysis made purely scientific purposes.<sup>a</sup>

<sup>a</sup> For a further presentation of this subject from the view point of an experienced petrographer see *Washington, U. S., Manual of the Chemical Analysis of Rocks*, 1904, pp. 8-17.

The importance of the points indicated in the foregoing paragraph is shown by the difference between the analyses given below. The specimens were taken and analyzed at widely separated times and by different persons, it is true, but they were unquestionably from the same rock mass, in which, however much the relative proportions of the different mineral constituents might vary within certain limits, there can be no reason to doubt the general distribution of all the elements shown by the second analysis.

*Analyses of specimens taken from the same rock mass at different dates.*

	Earlier analysis.	Later analysis. <sup>a</sup>		Earlier analysis.	Later analysis. <sup>a</sup>
SiO <sub>2</sub> .....	54.42	53.70	Li <sub>2</sub> O.....	Trace.	Trace.
TiO <sub>2</sub> .....		1.92	H <sub>2</sub> O below 110°.....		.80
Al <sub>2</sub> O <sub>3</sub> .....	13.37	11.16	H <sub>2</sub> O above 110°.....	c 2.76	2.61
Cr <sub>2</sub> O <sub>3</sub> .....		.04	CO <sub>2</sub> .....	1.82	
Fe <sub>2</sub> O <sub>3</sub> .....	b 6.61	3.10	P <sub>2</sub> O <sub>5</sub> .....		1.75
FeO.....	b 3.52	1.21	SO <sub>3</sub> .....		.06
MnO.....		.04	F.....		.44
CaO.....	4.38	3.46	Cl.....		.03
SrO.....		.19			
BaO.....		.62		99.58	100.40
MgO.....	6.37	6.44	Less O for F.....		.19
K <sub>2</sub> O.....	10.73	11.16			
Na <sub>2</sub> O.....	1.60	1.67			100.21

<sup>a</sup> A still more recent analysis of another of the series of rocks of which this is an example has shown that this "later analysis" is itself probably incomplete and incorrect in part—incomplete because of the probable presence of 0.2 per cent or more of ZrO<sub>2</sub>, incorrect because of the error in Al<sub>2</sub>O<sub>3</sub> resulting from having counted the ZrO<sub>2</sub> as Al<sub>2</sub>O<sub>3</sub>, and from the fact that titanium is not fully precipitable in presence of zirconium by Gooch's method (the one employed). This latter error involves both the TiO<sub>2</sub> and the Al<sub>2</sub>O<sub>3</sub>. (See § p. 115.)

<sup>b</sup> From the fact that repeated determinations of the iron oxides in this and related rocks from the same region show always a great preponderance of ferric oxide, it is not improbable that the figures given for the two oxides in the first analysis were accidentally transposed.

<sup>c</sup> In the published analysis it does not appear whether this is total water or, as seems probable, only that remaining above 100°.

Another instance of similar kind is given below. Here, again, certain differences are explainable by natural variations in the proportions of the constituent minerals, but it can hardly be doubted that TiO<sub>2</sub>, BaO, SrO, P<sub>2</sub>O<sub>5</sub>, and SO<sub>3</sub> were present in both specimens in approximately the same amounts. In the earlier analysis determinations of some supposedly unimportant constituents were purposely omitted, or made only qualitatively, with results that can not be otherwise than fatal to a full comprehension of the mineralogical nature of the rock.

*Analyses of specimens taken from the same rock mass at different dates.*

	Earlier analysis.	Later analysis.		Earlier analysis.	Later analysis.
SiO <sub>2</sub> .....	44.31	44.65	Na <sub>2</sub> O.....	4.45	5.67
TiO <sub>2</sub> .....	Not est.	.95	Li <sub>2</sub> O.....		Trace.
Al <sub>2</sub> O <sub>3</sub> .....	17.20	13.87	H <sub>2</sub> O below 110°.....	.77	.95
Fe <sub>2</sub> O <sub>3</sub> .....	4.64	6.06	H <sub>2</sub> O above 110°.....		2.10
FeO.....	3.73	2.94	H <sub>2</sub> O by ignition.....	3.30	
MnO.....	.10	.17	CO <sub>2</sub> .....		.11
CaO.....	10.40	9.57	P <sub>2</sub> O <sub>5</sub> .....		1.50
SrO.....		a .37	Cl.....		Trace.
BaO.....		.76	SO <sub>3</sub> .....		.61
MgO.....	6.57	5.15			
K <sub>2</sub> O.....	3.64	4.49		99.11	99.92

<sup>a</sup> Not entirely free from CaO.

Prof. F. W. Clarke has shown that the combined percentages of titanic and phosphoric oxides in rocks of the earth's crust, averaged from hundreds of analyses, amount to 0.8 per cent. When the determination of these is neglected the error falls on the alumina. If the alumina is then used as a basis for calculating the feldspars, it is easy to see that a very large average error in the latter may result, amounting to several per cent of the rock.

In order to emphasize more strongly the importance of completeness in analysis, a few facts brought out by the hundreds of rock analyses made in this laboratory may be cited. It has been demonstrated most conclusively that barium and strontium are almost never-failing constituents of the igneous rocks of the United States and of many of their derivatives. The amounts are usually below 0.1 per cent for each of the oxides of those metals, but higher amounts are by no means uncommon. Furthermore, the weight of barium is almost without exception in excess of that of strontium; but a still more important point is that the igneous rocks of the Rocky Mountain region, so far as examined, show far higher average percentages of both metals than the rocks from the eastern and the more western portions of the United States. The following examples serve to illustrate certain types of Rocky Mountain igneous rocks: Of seven rocks forming a Colorado series, six held from 0.13 to 0.18 per cent of BaO, while in the seventh the percentage was 0.43. The SrO ranged from 0.07 to 0.13 per cent for six and was 0.28 for that one highest in BaO. Of thirteen geologically related rocks from Montana, embracing basic as well as acidic and intermediate types, the range of BaO was from 0.19 to 0.37 per cent, with an average of 0.30 per cent. Three others of the same series contained 0.10 per cent or less, while the seventeenth carried 0.76 per cent BaO. The SrO ranged from 0.37 per cent in the last instance to an average of 0.06 for the other sixteen. Certain peculiar rocks from Wyoming carry from 0.62 to 1.25 per cent BaO and from 0.02 to 0.33 per cent SrO. Surely this concentration of certain chemical elements in certain geographic zones has a significance which future geologists will be able to interpret if those of to-day are not.

Again, vanadium is an element which few chemists have ever thought of looking for in igneous rocks, though it has long been known to occur in magnetites and other iron ores. A. A. Hayes, in 1875, reported its occurrence in a great variety of rocks and ores. To quote from Thorpe's Dictionary of Chemistry: "It is said to be diffused with titanium through all primitive granite rocks (Dieulafait), and has been found by Deville in bauxite, rutile and many other minerals and by Bechi and others in the ashes of plants and in argillaceous limestones, schists, and sands." It is further reported to

comprise, as the pentoxide, up to 0.1 per cent of many French and Australian<sup>a</sup> clays, 0.02 to 0.03 per cent of some basalts, 0.24 per cent of a coal of unknown origin, and 0.45 per cent of a coal from Peru. Still later examinations in this laboratory of about 100 rocks, chiefly igneous, covering nearly the whole continental territory of the United States, show not only its general qualitative and quantitative distribution, but that it predominates in the less siliceous igneous rocks and is absent, or nearly so, in those high in silica. In some of the more basic rocks it occurs in sufficient amount to affect seriously the figures for the oxides of iron unless separately estimated and allowed for (see  $\beta$ , p. 140), a matter of considerable importance, since the petrographer lays great stress on accuracy in their determinations.

The same investigation has also thrown some light on the distribution of molybdenum, which seems to be confined to the more siliceous rocks and to occur in quantities far below those commonly found for vanadium.

Finally, had it not been my practice of late years to look for sulphur in rocks, even when no sulphides were visible to the eye, its almost invariable presence in the form of sulphide and consequent connection with the long mystifying lack of agreement between results for ferrous iron obtained by the Mitscherlich and the hydrofluoric-acid methods might not have been suspected (see A, p. 131).

While strongly upholding the necessity for more thorough work, necessarily somewhat at the expense of quantity, it is far from my intention to demand that an amount of time altogether disproportionate to the immediate objects to be sought should be expended on every analysis. But it is maintained that, in general, the constituents which are likely to be present in sufficient amount to admit of determination in the weight of a sample usually taken for analysis—say 1 gram for  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , etc., to 2 grams for certain other constituents—should be sought for, qualitatively at least, in the ordinary course of quantitative work, and their presence or absence noted among the results. If present in little more than traces, that knowledge alone may suffice, for it is often more important to know whether or not an element is present than to be able to say that it is there in amount of exactly 0.02 or 0.06 per cent. In the tabulation of analyses a special note should be made in case of intentional or accidental neglect to look for substances which it is known are likely to be present. Failure to do this may subject the analyst to unfavorable criticism when, at some future time, his work is reviewed and the omissions are discovered by new analyses.

Finally, whenever possible, a thorough microscopical examination of the rock in thin section should precede the chemical analysis. This

<sup>a</sup> J. C. H. Mingaye has lately confirmed its wide distribution in Australian rocks, coals, etc. (Records Geol. Survey New South Wales, vol. 7, pt. 3, 1903, p. 213.)



may be of the greatest aid to the chemist in indicating the presence of unusual constituents or of more than customary amounts of certain constituents, whereby, possibly, necessary modifications in the analytical procedure may be employed without waste of time or labor.<sup>a</sup>

If the point be raised that many of the published analyses emanating from the Survey laboratories, even my own earlier ones, are not in accord with the advocacy of completeness contained in the foregoing pages, it may be remarked that these ideas have been to a considerable degree evolved during a personal experience of over twenty-five years in this line of work, and that frequently the exigencies were such as to compel restriction in the examination. Where the latter has been the case, subsequent developments have in some cases shown it to be bad policy in every respect. It is better, for both the geologist and the chemist, to turn out a limited amount of thorough work than a great deal of what may ultimately prove to be of more than doubtful utility.

## 2. THE CONSTITUENTS OCCURRING IN SILICATE ROCKS.

It is to the decomposition of the original igneous rocks or their magmas and their derivatives that nearly all ore bodies in the United States owe their origin by one or another process of concentration. A certain class of concentrations probably separated from magmas in the fluid state before solidification. Hence it is the natural and inevitable inference that sufficiently careful examination of these rocks would show them to contain all or nearly all the known elements, not necessarily all in a given rock, but many more than anyone has yet found.<sup>b</sup> Mechanical and analytical difficulties have thus far stood in the way of experimental proof that this is so, owing to the great scarcity of many of the elements. In exceptional cases it may be desirable to subject a given material to very exhaustive analysis, as, for instance, when searching for the origin of ore deposits. Ordinarily, however, the demands of the petrographer and geologist are satisfied with a knowledge of the quantitative relations of those constituents which can be determined in a limited amount of the sample—say from one-half gram to 2, or occasionally 5, grams. In general the discussion relating to silicate analysis in this volume will be confined to such separations as may be required in the analysis of an igneous, metamorphic, or sedi-

<sup>a</sup> See also Washington, H. S., *Chemical Analysis of Rocks*, 1904, pp. 6-7. The foregoing tables and accompanying remarks, including several sentences preceding the tables, have been largely taken from my paper entitled *A plea for greater completeness in chemical rock analysis*: *Jour. Am. Chem. Soc.*, vol. 16, 1894, pp. 90-93; *Chem. News*, vol. 69, 1894, p. 163. See also *Distribution and quantitative occurrence of vanadium and molybdenum in rocks of the United States*: *Am. Jour. Sci.*, 4th ser., vol. 6, 1898, p. 20; *Chem. News*, vol. 78, 1898, p. 216; and *Bull. U. S. Geol. Survey* No. 167, 1900, p. 49.

<sup>b</sup> F. Sandberger's researches have shown to what extent this is true of a large number of those elements contributing to the filling of metalliferous veins, and L. Dieulafoy by his elaborate qualitative researches showed how universal was the distribution of copper, zinc, barium, strontium, etc., in the primordial rocks.

mentary rock of complex mineralogical composition, in which the majority and possibly all of the ingredients in the list given below may occur in weighable or readily discoverable quantities:

SiO<sub>2</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, V<sub>2</sub>O<sub>5</sub>, FeO, MnO, NiO, CoO, MgO, CaO, SrO, BaO, ZnO, CuO, K<sub>2</sub>O, Na<sub>2</sub>O, Li<sub>2</sub>O, H<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, S,<sup>a</sup> SO<sub>2</sub>, C,<sup>b</sup> CO<sub>2</sub>, F, Cl, N.

To the above list might be added certain others, as the group of so-called rare earths, besides tin, platinum, tantalum, columbium, boron, glucinum, helium. Some of these occur at times in determinable amounts, though in my experience tantalum, columbium, and glucinum have not been met with. They may very well have been overlooked by reason of the absence of distinctive tests for identification. Thorium, cerium, and other rare earths are probably more common as constituents of silicate rocks than has been generally supposed. Their presence and amount can be so readily and certainly detected by the methods given in their proper place that the reason for neglecting to look for them is no longer so strong as it used to be, especially when there is microscopic or other evidence of the presence of minerals likely to contain them.

### 3. THE DISTRIBUTION AND OCCURRENCE OF CERTAIN MINOR CONSTITUENTS.

For the suggestion of this section and for much of its contents I am in no small degree indebted to Doctor Washington's book (*Manual of the Chemical Analysis of Rocks*, pp. 18-21), already more than once referred to.

Doctor Washington says:

"The increased number of analyses of igneous rocks, especially of unusual types, and the more frequent determination of the minor constituents, with the vast mass of data obtained by the use of the microscope, have shown that certain of the rare elements are prone to occur in rocks of certain chemical characters. While our knowledge along this line is far from complete, a few words may be devoted to this subject, as it will often be of use to the analyst to know which elements should be especially looked for and which may be safely neglected."

*Titanium*.—Not long ago held to be rare, this element is now recognized to be one of the most universally distributed and about tenth in actual abundance in the explored crust of the earth. So far as my experience goes, it is entirely absent from no igneous, metamorphic, or sedimentary rock of a more or less siliceous character. The recognition of this fact has been rendered possible and easy by its characteristic behavior, when in solution, toward hydrogen peroxide. Though

<sup>a</sup> Usually as pyrite, occasionally as lazurite, not infrequently as pyrrhotite.

<sup>b</sup> As graphite or coal matter.

seemingly present even in the most siliceous rocks, it is most abundant in the so-called basic ones. Its chief mineral occurrences are rutile, octahedrite or anatase, ilmenite, titanite, and perovskite, but it is also a component in smaller amounts of many pyroxenes, hornblendes, biotites, and garnets. Owing to the refractory nature of some of its compounds it tends to concentrate in the residual products of decomposition of many rocks; hence its high percentage in clays of a class. It is very unusual to find titanium present in amounts that can properly be designated only as traces. In the great majority of cases its amount will not exceed 1 per cent, but it may rise to over 5 per cent.

*Zirconium*.—The chemically related element zirconium is likewise now known to be more widely distributed than was formerly supposed, but it is much less common than titanium, rarely making up 0.2 per cent of a rock and being usually under 0.05 per cent. "It is most apt to occur in granites, rhyolites, syenites, and in nephelisyenites, phonolites, tinguaite, and tephrites, and is most abundant in those which are high in soda, such as the last four. It is rarely met with in the more basic rocks, especially those rich in lime, magnesia, and iron. Zirconium is usually found as the silicate zircon, especially in granites and syenites, but is also an ingredient of the rare minerals eudialyte, lävenite, and rosenbuschite."

*Chromium*.—"Chromium is almost wholly confined to the basic rocks, especially those which are high in magnesia and low in silica and consequently contain abundant olivine, such as peridotite and dunite. It occurs as chromite and picotite (chrome-spinel), and in some augites, biotites, and olivines. It may occur up to one-half of 1 per cent of  $\text{Cr}_2\text{O}_3$ ."

*Vanadium*.—The distribution of this element has been in part covered by the remarks on pages 18–19. To leave out of account the well characterized mineral vanadates, its most stable and normal form in the silicates seems to be that of trivalency, corresponding to the oxide  $\text{V}_2\text{O}_5$ , in which form it replaces alumina and possibly ferric oxide to a very limited extent—a few hundredths of one per cent—in pyroxene hornblendes, and biotites. It hence predominates in the less siliceous igneous rocks. As the mineral roscoelite, essentially a vanadic alumino-potassic silicate, or some other similar silicate, it is rather widely distributed as a component of certain sandstones in western Colorado and eastern Utah, sometimes in large amount.<sup>a</sup> It may well exist in a similar condition in clays and the ashes of coals. The conclusion being based on the above observations, it should not occur as constituent of nonaluminous and nonferrie rock-forming minerals.

<sup>a</sup> Hillebrand, W. F., and Ransome, F. L., *Am. Jour. Sci.*, 4th ser., vol. 10, 1900, p. 120; *Bull. U. S. Geol. Survey* No. 262, 1905, p. 9.

as to olivine, at least, this conclusion has been found to be justified. Vanadium is also a constituent of ilmenite in titaniferous iron ores. The existence of the copper sulphovanadate, sulvanite, and the occurrence of vanadium in apparent combination with sulphur in certain peculiar carbonaceous ores of eastern Utah and Peru<sup>a</sup> indicate a possible wider range of combination in exceptional cases.

*Manganese.*—This element is found in ferromagnesian minerals in nearly all rocks, though as the result of their alteration it may sometimes appear, particularly on surfaces of limestones and sandstones, in a more or less peroxidized condition. Its amount will rarely exceed .3 per cent. I fully agree with Doctor Washington in regarding the high figures commonly reported as due to analytical error. There is no element which the average chemist is more prone to report too high, by gravimetric methods.

*Nickel and cobalt.*—These elements are found in the olivine of peridotite rocks and also in pyrite and pyrrhotite, hornblende, and magnetite. Even in peridotites the percentage of nickel will seldom exceed 0.1 per cent, while that of cobalt rarely if ever exceeds a trace. As with manganese, the percentage of nickel has often been reported too high.

*Copper.*—Owing to the extreme precautions necessary to exclude its introduction from utensils and reagents during analysis, there is less positive information regarding the distribution of copper than of some other elements which do not exceed it in amount. Nevertheless, there is reason to believe that it is almost if not quite as universally distributed as most of the other minor constituents. Its particular home appears to be in diabase, gabbro, amphibolite, and other basic rocks with pyroxene and amphibole. J. B. Harrison, in a recent report (1906) to the Science and Agriculture Department of British Columbia, shows its occurrence in hundredths of 1 per cent in many igneous rocks of that colony. So far as my experience goes, it can be found almost invariably, if looked for, in the rock analyses carried out in the Survey laboratory. But for the reason above given it is seldom reported unless extra precautions have been taken to prevent its extraneous entry into the analysis.

*Barium and strontium.*—Feldspathic rocks are those most likely to carry these elements, the former almost always in excess of the latter. According to Washington, there is considerable evidence, in part unpublished, that barium is apt to be most abundant in rocks which are high in potassium. It occurs "in orthoclase (as the hyalophane molecule) and possibly also in labradorite and anorthite (as celsian) as well as in a few biotites and muscovites." It has been found in a few peculiar rocks in excess of 1 per cent, in terms of the oxide, but in the greater part of the silicate rocks of the United States its percent-

<sup>a</sup> As to the Peruvian occurrence see Hewett, Foster, Eng. Min. Jour., vol. 32, 1906, p. 385, and Bravo, José J., Bol. Soc. Ing. (Lima), vol. 8, 1906, p. 171.

age is well below 0.2. Strontium has been found as high as 0.3 to 0.4 per cent, but generally there is little more than a trace of it.

*Lithium.*—Although one of the most universally distributed of the elements, it is almost never met with in rocks in more than spectroscopic traces. Aside from the lithium minerals lepidolite and spodumene, it is found in the alkali feldspars, in muscovite, beryl, and other minerals. According to Washington, there is reason for the belief that it is especially prone to occur in highly sodic rocks.

*Phosphorus.*—This element is found in greatest abundance in the more basic igneous and metamorphic rocks, and is practically never entirely absent. It especially affects those rocks "which are high in lime and iron rather than in magnesia." Its chief mineral occurrence is in apatite, though it may be found in xenotime and monazite. While the percentage is usually well under 1, it may considerably exceed this figure.

*Sulphur.*—Aqueous extraction of a powdered rock will in many cases remove traces of sulphur and chlorine. These are to be regarded in almost every case rather as derived from infiltrating waters than as products of decomposition of the constituents of the rock itself. Essential rock-forming minerals containing sulphur in the oxidized condition are limited to hauynite and noselite, minerals which are found chiefly in the more basic rocks, and especially those high in soda. In the sulphide condition the element is of very wide distribution, both as pyrite and pyrrhotite, less often as chalcopyrite and possibly other sulphides, also in the mineral lazurite, and here too in greater abundance in the basic rocks. It is a very common error of most chemists to report the sulphur in silicate and carbonate rocks, clays, etc., as  $\text{SO}_3$  instead of S. It may now and then occur in both states, but much more often only in the sulphide condition.

*Chlorine.*—"Chlorine is present most abundantly in rocks which are high in soda, and especially when so low in silica that nephelinite is present, though it is also found sometimes in nephelinite-free rocks, and in a few cases in quartz-bearing ones. It is an essential component of sodalite [also noselite —W. F. H.], and is also present in scapolite and in a few apatites." Its amount when present rarely exceeds 0.2 or 0.3 per cent. When seeking for the chlorine of these minerals it is always well to extract the powder first with cold water (see preceding paragraph). It is to be remembered that fluid inclusions in minerals sometimes contain sodium chloride, which would be largely extracted from the powder by water.

*Fluorine.*— "Fluorine seems to have no special preference as to magma, though, on the whole, it is found more frequently in acid than in basic rocks. It is also apparently most apt to be met with as fluorite in rocks containing nephelinite, as foyaites and tinguaites. It is an essential constituent of fluorite and most apatite, and as an integral

part of the last mineral is almost universally present. It also occurs in biotites and other micas, in some hornblende and augite, as well as in tourmaline, topaz, chondrodite, etc." When determined it is usually reported in amounts under 0.1 per cent, but freedom of the weighed calcium fluoride from contamination being assumed, the reported amount is invariably too low because of the inaccuracy of the method for its separation (see c, p. 158).

*Other minor constituents.*—"Glucinum, as a component of beryl, is most frequent in granites, pegmatites, and quartzose gneisses. Tin is confined to the acid rocks, granite, quartz porphyry, and rhyolite, and its presence is due [often—W. F. H.] to pneumatolytic processes. It occurs as cassiterite, and in traces in ilmenite, micas, and feldspars. The rare-earth metals occur in allanite, xenotime, monazite, and other minerals of even greater rarity, and seem to be especially frequent in acid rocks and possibly those with much soda. Molybdenum tungsten, and uranium are almost exclusively confined to the very siliceous rocks. Zinc has been met with in granite, as well as in basic rocks, but no generalization in regard to it is possible as yet [nor in regard to lead, which can often be found by using considerable amounts of rock material, as in some of the rocks of Leadville, Colo., and in those of British Guiana.—W. F. H.] Platinum is found almost exclusively in peridotites, but is occasionally met with in connection with gabbros. Boron, as a constituent of tourmaline, is most apt to occur in highly siliceous rocks." Boron is also common in many contact-metamorphic schists. Gold and silver have been found repeatedly. According to Harrison,<sup>a</sup> some of the British Guiana rocks contain gold enough to account for the commercially valuable deposits in residual soils resulting from the rock decomposition. Tantalum and columbium occur in some granites and pegmatites, but have never been reported in percentages in rock analysis, so far as known to me.

#### 4. SUMMATION OF ANALYTICAL RESULTS AND LIMITS OF ALLOWABLE ERROR.

As is well known, a complete silicate-rock analysis which foots up less than 100 per cent is generally less satisfactory than one which shows a summation somewhat in excess of 100. This is due to several causes. Nearly all reagents, however carefully purified, still contain, or extract from the vessels used, traces of impurities, which are eventually weighed in part with the constituents of the rock. The dust entering an analysis from first to last is considerable, washings of precipitates may be incomplete, and if large filters are used for small precipitates the former may easily be insufficiently washed.

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<sup>a</sup>Rept. Sci. and Agr. Dept. British Guiana, 1906.

Given the purest obtainable reagents, an ample supply of platinum, facilities for working, and a reasonably clean laboratory, there is usually little excuse for failure on the part of a competent chemist to reach a summation within the limits 99.75 and 100.50. Failure to attain 100 per cent in several of a series of analyses of similar nature should be the strongest evidence that something has been overlooked. Excess above 100.5 per cent should be good ground for repeating portions of the analysis in order to ascertain where the error lies, for it is not proper to assume that the excess is distributed over all determined constituents. It is quite as likely, in fact more than likely, to affect a single determination and one which may be of importance in a critical study of the rock from the petrographic side.

Doctor Washington would extend the allowable limits above given to 99.50 and 100.75. If the analysis has to be made in other vessels than those of platinum a higher limit than 100.50 is certainly called for, but it is extremely doubtful if a lower one than 99.75 should be accepted, for, as has been said, there is a tendency toward too high results in any case, and a summation of less than 99.75 with vessels other than platinum would be indicative of rather gross error on the part of the analyst.

As to the allowable variation in duplicate determinations of the same constituent no rigid rules can be laid down. Doctor Washington has proposed the following,<sup>a</sup> with which the independent experimental values of Doctor Dittrich<sup>b</sup> are in substantial agreement: For  $\text{SiO}_2$  and others which amount to 30 per cent and over, from 0.2 to 0.3 per cent; for  $\text{Al}_2\text{O}_3$  and others which amount to from 10 to 30 per cent, 0.1 to 0.2 per cent; for constituents which amount to from 1 to 10 per cent, 0.05 to 0.1 per cent. These percentages are in terms of the whole rock and not of the particular constituent.

While it is desirable sometimes, and for the beginner imperative, to make duplicate determinations, agreement is not to be taken as proof that the results are correct unless they have been arrived at by different methods.

Too great stress can not be laid upon the importance, especially for the analyst of limited experience, of consistently testing most of his final precipitates and filtrates as carefully as possible in order to be sure, on the one hand, that the substances reported by a given name are wholly such and, on the other, that all is reported. In no other way can an analyst so soon acquire the needed confidence in himself and his methods.

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<sup>a</sup> Manual of the Chemical Analysis of Rocks, 1904, p. 24.

<sup>b</sup> Neues Jahrb. f. Min. u. Geol., vol. 2, 1903, p. 69.

## 5. STATEMENT OF ANALYSES.

Until recently it has been the practice in this laboratory to tabulate the constituents of a rock somewhat in the order of their determination, beginning with  $\text{SiO}_2$  as the chief constituent and grouping together all chemically related oxides, as shown, for instance, on page 17.

From a strictly scientific point of view a chemical classification founded on a separation into basic and acidic atoms or radicals would be more satisfactory, but until we learn to find out what silicic radicals are present and in what relative amounts, also how much free silica there may be, it is useless to think of employing the arrangement so valuable in stating water analyses.

Of late, petrographers have begun to demand, with considerable reason, an arrangement "which shall bring the essential chemical features—both the percentage figures and the molecular ratios—prominently and compactly before the eye, so that the general chemical character and the relations of the various constituents may be seen at a glance."<sup>a</sup>

In accordance with this demand it is now our practice to follow pretty closely the arrangement proposed by Pirsson and later strongly advocated by Washington,<sup>b</sup> namely:

$\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{MgO}$ ,  $\text{CaO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{H}_2\text{O}$  (above 105–110°),  $\text{H}_2\text{O}$  (below 105–110°),  $\text{CO}_2$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{SO}_3$ ,  $\text{Cl}$ ,  $\text{F}$ ,  $\text{S}$  ( $\text{FeS}_2$ ),  $\text{Cr}_2\text{O}_3$ ,  $\text{V}_2\text{O}_5$ ,  $\text{NiO}$ ,  $\text{CoO}$ ,  $\text{CuO}$ ,  $\text{MnO}$ ,  $\text{SrO}$ ,  $\text{BaO}$ ,  $\text{Li}_2\text{O}$ ,  $\text{C}$ ,  $\text{NH}_3$ .

By this arrangement the nine constituents which in the great majority of cases determine the character of the rock are placed at the head of the list, thus greatly facilitating the comparison of different analyses similarly arranged, especially when, as Washington recommends, the molecular ratios are calculated for these leading constituents and placed immediately after the corresponding oxides. The order of the remaining members is determined somewhat by the following considerations:  $\text{CO}_2$  is placed next after  $\text{H}_2\text{O}$ , since these two are generally a measure of the alteration the rock may have undergone.  $\text{TiO}_2$  and  $\text{ZrO}_2$  naturally follow  $\text{CO}_2$  on chemical grounds, and  $\text{SO}_3$  and  $\text{Cl}$ , being common constituents of the sodalite group, are conveniently placed together.

It may be said with regard to the use of the word "trace" that the amount of a constituent thus indicated is supposed to be below the limit of quantitative determination in the amount of the sample taken for analysis. It should in general, for analyses laying claim to completeness and accuracy, be supposed to indicate less than 0.02 or even 0.01 per cent.

<sup>a</sup> Washington, H. S., The statement of rock analyses: Am. Jour. Sci., 4th ser., vol. 10, 1900, p. 61.

<sup>b</sup> Loc. cit.



## 6. TIME NEEDED FOR MAKING AN ANALYSIS.

The question has often been put, "How long does it take to complete an analysis of this kind?" This will depend, of course, on the mineral complexity of the sample and on the personal factor of the individual worker. If there is a competent assistant to do the grinding, and specific-gravity determinations are not required, it is quite possible after long experience for a quick worker to learn to so economize every moment of time in a working day of seven hours, with an abundance of platinum utensils and continuous use of air and water or steam baths through the night, as to finish every three days, after the completion of the first analysis, barring accidents and delays, one of a series of rocks of generally similar character, each containing from eighteen to twenty quantitatively determinable constituents excluding, for instance, fluorine, carbon as such, nitrogen, metals of the hydrogen sulphide group, and cobalt. But such an output of work implies an unusual freedom from those occasional setbacks to which every chemist is exposed.

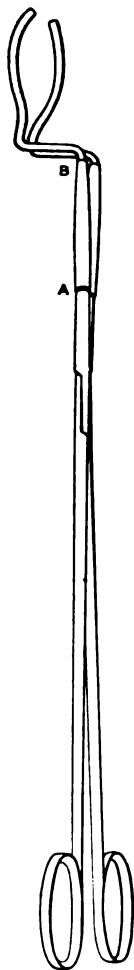


FIG. 1. Platinum-tipped crucible tongs. The parts A, B, also of heavy platinum, are hollow, to serve as sockets for the cheaper metal of the handles.

## 7. USEFUL APPLIANCES AND APPARATUS.

In connection with the foregoing remarks it is in place to mention a few aids to the chemist which are in constant use in this laboratory and have come to be well-nigh indispensable. None is novel in principle and all are in use elsewhere, but some are not so commonly known as they deserve to be, hence this allusion to them. Certain cheap and simple forms of colorimeters have been found very useful, and since they are adapted to the determination of a variety of substances their description finds most appropriate place here.

## A. CRUCIBLE TONGS.

Fig. 1 represents a form of platinum-tipped crucible tongs devised by Dr. A. A. Blair many years ago. With them a crucible can be securely grasped and brought into any desired position while still hot. To the contents, if in fusion over the blast flame, can be imparted the

rotatory motion so often desirable. Above all, the cover need not be in the slightest degree displaced, as when using the common form of platinum-tipped tongs.

#### B. RADIATORS FOR VOLATILIZING LIQUIDS AND SOLIDS.

Fig. 2 represents a very useful adjunct to the worktable and especially to the draft cupboard, whereby the liquid contents of crucibles can be speedily evaporated at almost any desired temperature and the

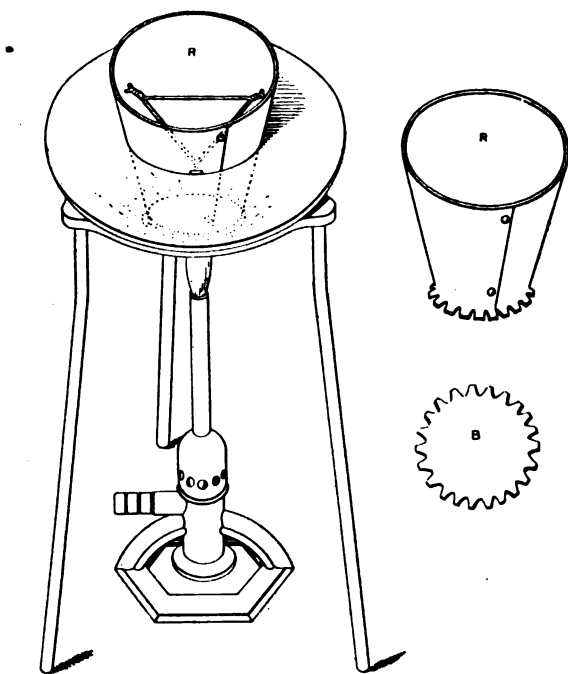


FIG. 2.—Radiator for rapid and safe evaporation. R is of sheet iron or aluminum, also nickel (Jannasch). A convenient height is 7 cm., width at top 7 cm. and at bottom 5 cm. The base, B, may be of iron, nickel, or platinum, but not aluminum, which will not stand the temperature of the direct flame. Platinum is most satisfactory by reason of its long life and radiating power. The manner of attaching B to A is by turning the cogs of B up and over those of A. Evaporation may be greatly hastened when desired by placing upon A a cast-iron ring with its opening somewhat larger than the mouth of the underlying crucible.

dehydration of many solids effected much more safely than on an iron plate or sand bath. I do not recall who originated this form of air bath, but it has been in use here for over twenty years and is identical in principle with the "Nickel-becher" of Jannasch. Nickel undoubtedly has a certain advantage in not rusting as does iron, but the form depicted in fig. 2 can easily be made anywhere of sheet iron riveted at the joint, the bottom being securely held by a notched flange at the extremity of the truncated cone. A crucible placed on the platinum triangle becomes uniformly heated by hot air, and large quantities of

liquid, even sulphuric acid, can be thus volatilized in a short time without ebullition or spattering. The life and effectiveness of this appliance is greater if the bottom is of sheet platinum, but if an iron

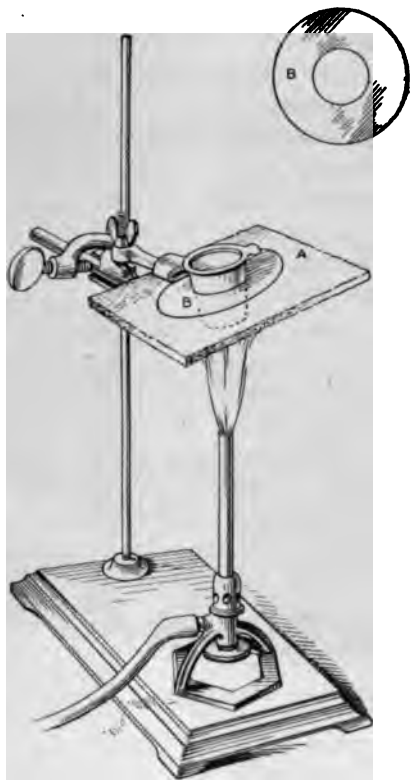


FIG. 3.—Device for excluding flame gases from the interior of crucibles during ignition. A, Asbestos board; B, stiff platinum foil of size suitable to the crucible used and having an opening to admit the crucible to about two-thirds of its depth. This disk may be used alone upon the ring of an ordinary stand and in horizontal position, or, preferably as shown in the figure, in combination with the asbestos board, in which is an opening considerably larger than the crucible. The combination should then be given the inclined position shown, so as to allow the products of combustion to flow to one side without the possibility of their enveloping the mouth of the crucible. In the lack of platinum a perforated asbestos board alone will yield fair service with a blast, but not as a rule with an ordinary burner.

bottom wears out it is usually possible to replace it one or more times before the sides fail. A coating of aluminum paint on the sides adds to their life.

By an extension of the principle illustrated in fig. 2 very considerable amounts of ammonium salts and other easily volatile solids may be driven off from platinum dishes with little danger of loss by spattering and none by overheating. The dish containing the dry or nearly dry matter is placed in another of such size that the bottom of the inner dish is at some distance from that of the outer.— The outer dish may be of iron, direct contact between it and the inner dish being prevented by wisps of asbestos overlapping the edge of the former, but the excellent conductivity and radiation of platinum recommends it above anything else. Aluminum will not stand the application of a full flame. If the contents of the dish are moist at first, a low flame will soon dry them, when the heat may be increased at will and the operation allowed to proceed without supervision. A sand bath may sometimes be substituted for the radiator.

#### C. PERFORATED DISK FOR CRUCIBLES.

Fig. 3 shows an arrangement for the ignition of crucibles when it is desired to exclude flame gases from their interior, as in the ignition of ferric oxide and the determination of sulphur by fusion with an

alkali carbonate. The original idea of J. Löwe<sup>a</sup> was to use perforated clay disks in determining the ash of coals. Later, Lunge and others advocated asbestos board, but this has the disadvantage that it lasts but a short time and the fibers adhere to the crucible to some extent. By neither of these is it easy to secure a very high temperature in the crucible. Therefore a disk of platinum is preferable because of its durability, cleanliness, and high conducting power, though its cost may preclude having a set to fit different sizes of crucibles. The platinum disk and asbestos board may be conveniently used in combination, as in the figure. The asbestos then has a large hole, over which a smaller perforated disk of platinum is laid.

#### D. WEIGHING SCOOP OR TROUGH.

Fig. 4 represents a most convenient receptacle for the sample that is being weighed on the balance. It is made of platinum and is counterpoised by a leaden weight. It has the advantage over a watch glass in that its shape permits of ready introduction of its contents into narrow-mouthed receptacles when desired.

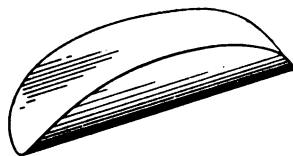


FIG. 4.—Weighing trough of platinum, to be balanced by counterpoise of lead. Length of trough about  $7\frac{1}{2}$  cm.; width before bending, 5 cm.

#### E. COLORIMETERS.

##### a. SURVEY FORM.

The Survey form of colorimeter shown in fig. 5 consists of two glass reservoirs and a darkened box. The glasses, G, may be of square or rectangular section, 8 to 12 cm. high and 3 to  $3\frac{1}{2}$  cm. inside measurement between those sides through which the liquid is to be observed.<sup>b</sup> These sides should, of course, be exactly parallel, the others need not be, but should be blackened externally. In order to exclude further the effect of side light, it is very convenient to have a simple light box, B, that can be easily held in the hand, stained black inside and out and with one end closed by a piece of ground glass, W, the other being open. For a space equal to the width of the glasses the cover is removed at the top next the glass end to permit the insertion of the glasses side by side in such a way that no light shall penetrate around their sides or between them. Immediately back of the glasses is a partition, P, with openings of appropriate size

<sup>a</sup> Zeitschr. anal. Chemis, vol. 20, 1881, p. 224.

<sup>b</sup> The allowable error in distance between the corresponding pairs of sides of the two glasses should not in any case exceed 1 per cent. Unfortunately there has been a disinclination on the part of dealers in this country to furnish glasses fulfilling this requirement, and held together by a durable cement which shall be proof against dilute sulphuric acid and alkali, though some have been obtained through Messrs. Eimer & Amend, of New York City. Canada balsam answers well for a time, but sooner or later it cracks, leaks then appear, and the sides soon drop off. It is, however, but a simple matter to cement them on again. But the use of Canada balsam is precluded with alkaline solutions, and hence for the chromium determination, as the solution at once becomes turbid.

cut in it. A stiffly sliding black cardboard shutter, S, is movable up and down immediately back of the partition, so that all light can be cut off except that which comes through the liquid. The ground-glass window, W, sliding up and down in slots, affords a much more uniform illumination than can be had without it.

Precautions of this kind are necessary if accurate results are to be counted on. Except for mere traces, this combination of glasses and darkened box insures greater accuracy and rapidity of work than Nessler tubes, and is preferable likewise, so far as my experience goes, to expensive instruments like the colorimeter of Soleil-Duboscq, etc.

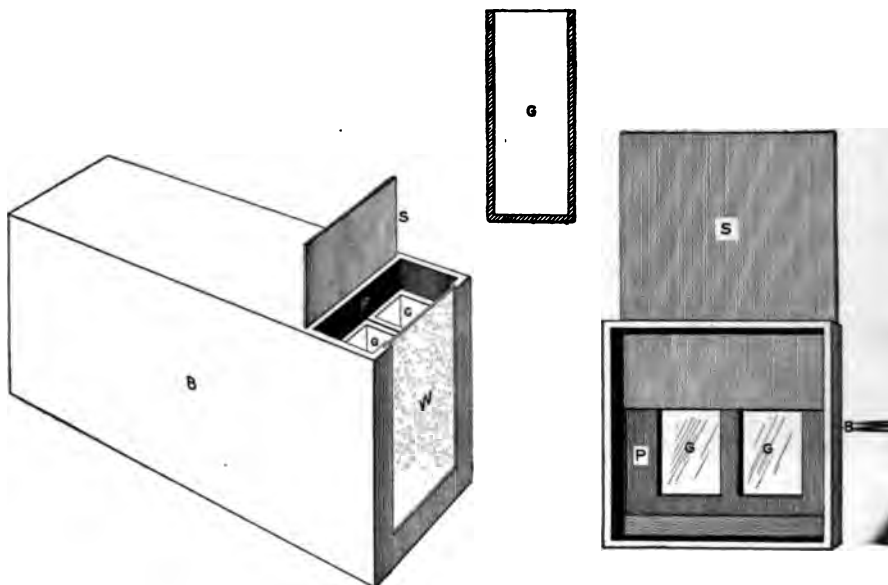


FIG. 5. —Survey form of apparatus for colorimetric determinations, in different aspects. G, One of two glasses of square or rectangular section, 8 to 12 cm. high and 3 to 3½ cm. inside measurement between those sides through which the liquid is to be observed. The other sides are blackened on the outside. B, Rectangular box about 35 cm. long and 12 cm. square, stained black inside and out, one end closed by a ground-glass window, W, the other open, and a portion of the top removed. P, Blackened partition, with openings corresponding to the interior dimensions of the glasses when in position. S, Blackened cardboard shutter sliding stiffly up and down back of the partition.

The manner of using the colorimeter is as follows: Any suitable amount of the standard solution, which is purposely made stronger than the solution to be tested, is placed in one of the glasses and an indefinite part or the whole of the test solution in the other. Water is then added from a burette to the standard until there is no distinction as to color. Perfect mixture is brought about by a glass rod flattened at one end. In making the color comparison the box is best held close to a window, so as to get a full, strong light. Daylight is far preferable to artificial light. The amounts of the substance to be determined and that in the standard cylinder are of course proportional to the volumes of the matched solutions.

## b SCHREINER'S FORM.

Dr. Oswald Schreiner, of the United States Department of Agriculture, has devised a colorimeter (fig. 6),<sup>a</sup> working on the same basic principle as the far more expensive one of Soleil-Duboscq, which has done good service in colorimetric work on phosphates, etc. Its description, personally communicated by Doctor Schreiner, is as follows:

The colorimeter in its simplest form consists essentially of graduated glass tubes, B, containing the standard and the unknown colorimetric solution, the column of the liquid in both tubes being changed by means of two smaller immersion tubes, A, also of glass. The upper ends of the tubes A are mounted in blocks of wood which fit into grooves on the body of the colorimeter. This permits the ready removal of the tubes from the colorimeter for cleaning when the liquids are changed. The graduated tubes, B, are supported by a block of wood about midway of the camera and are held in place by brass springs or clamps C, the tension of which can be regulated to suit the diameter of the tube, so as to allow it to be moved freely up or down by hand and yet be firmly held in position when the setting is made. It will be noticed from the figure that the glass tubes diverge slightly. This has been found of advantage, as it allows the operator to look down the center of both immersion tubes, as well as to bring the upper parts of the tubes, and consequently the images closer together. The openings to the immersion tubes can be covered by means of a microscope slide to prevent dust from falling into the tubes. The tubes are illuminated from the reflector, D, below, which carries a white sheet of cardboard or opal glass, and after passing through the tubes the light is reflected by a mirror, E, in the upper part of the instrument to the eye of the observer at F. The dimensions of the camera are about 70 by 32 by 16 cm. Standard glass slides may be inserted at G below one of the immersion tubes in place of the tube containing the standard solution, the immersion tube being retained so as to give similar images when viewed from above.

The measuring colorimeter tubes, B, are 25 cm. long, with inside diameter as nearly as practicable 27 mm.; in no case less than 25 mm. and not greater than 29 mm. The glass should be colorless and the bottoms well ground and polished, with internal surfaces of the bottoms plain, in no case appreciably convex or concave. The bottoms must be ground

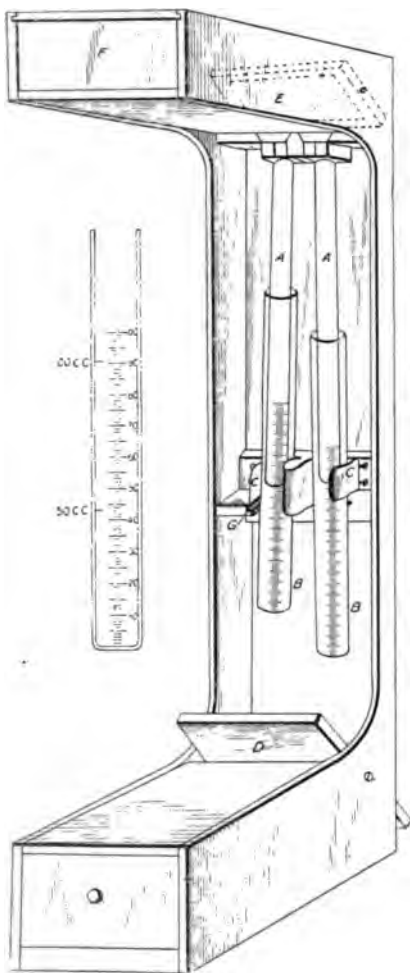


FIG. 6.—Schreiner's colorimeter.

<sup>a</sup> Jour. Am. Chem. Soc., vol. 27, 1905, p. 1192.

down sufficiently to make the ground surfaces a little larger than the internal diameters of the tubes; each of the tubes is provided with an etched scale of one hundred 2-mm. divisions. The scale begins at the level of the inner surface at the bottom, the length of the marks being 6 mm. and every fifth mark 12 mm., and every tenth mark numbered on the right side, 10, 20, 30, etc., beginning at the bottom. On the reverse side of each measuring tube may be etched two capacity marks, if desired, one for 50 cm.<sup>3</sup>, the other for 100 cm.<sup>3</sup>

The smaller tubes are likewise 25 cm. long, with outside diameter not greater than 20 mm. and not less than 18 mm., with the thickness of the glass the same in all the tubes. The bottoms of these tubes must be carefully ground and polished and the inside of the bottom never sensibly convex or concave. They must be of colorless glass with the bottoms so ground that the diameter of the ground portion exceeds the internal diameter of the tube. These tubes are to be provided with neither scale nor capacity marks.

The standard colorimetric solution is poured into one of the graduated tubes and put into place in the camera together with the immersion tube. The unknown colorimetric solution, made up to definite volume, is put into the other graduated tube and similarly placed in the instrument. The tube containing the solution of unknown strength is set at a convenient height, say fifty scale divisions, and the other tube containing the standard moved up or down, the operator watching the effect on the image in the mirror through the opening in the front of the camera. By moving the tube so that the image is alternately weaker or stronger than the standard, the setting can be accurately and quickly made. When both images show the same intensity of color, the setting is read by noting the division mark on the graduated tube opposite the ground bottom of the immersion tube. This reading will give the height of column of the standard which exactly corresponds with the intensity of color of the column of the unknown solution. It follows that the strengths of these two colorimetric solutions are inversely as the heights of the columns—that is, as the readings on the two tubes. If  $R$  is the reading of the standard solution of strength  $S$ , and  $r$  the reading of the colorimetric solution of unknown strength  $s$ , then  $s = \frac{R}{r} S$ . The same formula, of course, applies when the standard is fixed and the unknown solution moved up or down until the images are of equal intensity.

#### 8. PRELIMINARY QUALITATIVE ANALYSIS.

A complete qualitative analysis of a rock, preceding the quantitative examination, is in most cases a sheer waste of time. A few constituents may now and then be specially looked for, but in general time is saved by assuming the presence of most of them and proceeding on that assumption in the quantitative analysis.

This statement must not be misinterpreted, as has occurred, so as to convey the impression that qualitative analysis receives little consideration in the chemical work of the Survey. For ores, mine species, waters, etc., it is indispensable, as also in the testing of precipitates and filtrates during the course of quantitative analysis.

#### 9. QUALITY OF REAGENTS.

Unquestionably the greatest hindrance to good work in otherwise well-equipped laboratories is the difficulty in securing satisfactory reagents. This fact early became apparent to the Survey chemists, and it is mainly to their strenuous complaints and initiative that the present movement in favor of better quality is due. Much of

the glassware on the market is of a very inferior grade and utterly unsuited for analytical work.

All analyses performed in the Survey laboratory have been made with the purest reagents obtainable, either by purchase in the open market or by special preparation on the part of manufacturers or in the laboratory, but the most ceaseless vigilance has at all times to be practiced. As is now generally known, a "C. P." label is no guaranty whatever of the purity of a reagent. Every new purchase must be examined if it is one in which purity is a desideratum. In general, all so-called "C. P." chemicals should at least stand the tests laid down by Krauch.<sup>a</sup> The "guaranteed" reagents have been found at times to be worse than those emanating from sources which make no claim to special purity for their goods.

Acids made in this country can be had of a high grade, and these need no redistillation except for special experiments.

Hydrofluoric acid had to be always freshly distilled with potassium permanganate until the introduction of ceresin bottles afforded an article sufficiently pure for the most exacting work. Care must be taken to see that no particles of paraffin or ceresin are floating on the acid as it is poured out, and that this is free from traces of hydrochloric acid whenever it is to be used for attacking silicates with a view to determining chlorine. (C. b, p. 155). An important use for hydrofluoric acid is the decomposition of refractory silicates in the determination of ferrous iron. For this purpose it is necessary that it be free from any substance that will reduce permanganate. Sulphurous acid was found in one or two shipments, but this is a contamination against which care in manufacture can easily guard.

Ammonia ought always to be redistilled at short intervals, after first shaking up with slaked lime in order to decompose any ammonium carbonate that it may hold. Glass stock bottles may be coated inside with ceresin so as to prevent contact between the glass and the ammoniacal solution, a device that may be serviceable with other reagents.

Potassium bisulphate should be entirely free from silica, alumina, and heavy metals. The only correct way to get an accurate silica determination is to fuse the salt with a considerable amount of strong sulphuric acid, allow to cool, dissolve in water, and digest on the steam bath to coagulate the silica, which is then filtered; or, the salt may be dissolved in water and evaporated with a large excess of sulphuric acid till fumes of the latter rise copiously. On cooling, the mass is dissolved and treated as above. If a satisfactory article is not to be purchased, it can be made from equivalent parts of potassium sulphate and sulphuric acid. Even then the normal salt has first to be examined, for it has been found to contain

<sup>a</sup> Die Prüfung der chemischen Reagentien, 3d ed., Berlin, Julius Springer, 1896. English translation by J. A. Williamson, D. Van Nostrand Co., 1902.



notable amounts of lead, calcium, and silica. Sodium bisulphate is for some purposes preferable to the potassium salt. Both should, however, be converted to pyrosulphate by fusing in a large platinum dish and maintaining in fusion till all spattering has ceased and sulphuric acid rises copiously. The salt thus treated will not show the disagreeable frothing and spattering that is so objectionable when making fusions with the bisulphate and it is more quickly effective.

The phosphorus salt used for precipitating magnesium has been found to contain iron and silicon.

Ammonium oxalate almost always contains calcium, and must therefore be purified or specially prepared. The same may be said of oxalic acid. According to A. Classen,<sup>a</sup> ammonium oxalate sometimes contains lead.

Ammonium chloride has been known to contain manganese.

Hydrogen peroxide sometimes contains fluorine, which renders it unfit for use as a chemical reagent. The test for fluorine is carried out as follows: To 50 cm.<sup>3</sup> of the reagent add a slight excess of sodium carbonate and warm the solution. Filter if a precipitate forms, and to the boiling solution add calcium chloride in excess. Filter and gently ignite the precipitate. Treat it with dilute acetic acid, drop by drop, till the calcium carbonate is dissolved, filter, wash, gently ignite the residue, and test it with strong sulphuric acid in the usual way for fluorine. The perhydrol of E. Merck, now on the market, is a very pure and strong peroxide.

Sodium carbonate, one of the most important reagents in silicate analysis, can be had which does not carry more than  $2\frac{1}{2}$  milligrams of total impurity (see  $\beta$ , p. 72) in 20 grams (0.012 per cent). This should be reserved for the main portions, in which silica, alumina, etc., are to be determined. For other constituents, as phosphoric acid, fluorine, sulphur, a poorer grade is quite allowable, provided it is free from the element to be determined and from any other which might interfere with its determination. Silica is to be tested for by evaporating the solution of the carbonate or the dry salt with sulphuric acid in large excess till fumes of the acid are copiously evolved, dissolving the cooled mass in water, and digesting on the steam bath in order to coagulate the silica before filtering. Evaporation with hydrochloric acid will not yield all of the silica. Not long ago a very objectionable impurity in an otherwise acceptable article was encountered, namely, fluorine. The explanation of its presence is probably that cryolite had been used as the source of the carbonate. Sodium phosphate in small amounts seems to be a common contaminant of even good grades of sodium carbonate.

Sodium hydroxide made from sodium has been found with a large amount of lead, a fatal obstacle to successful mineral analysis, and also with small amounts of nickel.

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<sup>a</sup> Zeitschr. anal. Chemie, vol. 42, 1903, p. 518.

Owing to the solvent action on glass of many solutions of solid reagents, these solutions should be made up at frequent intervals in limited quantities, or, preferably, the solid should be dissolved as wanted. That this is no unnecessary refinement in really good work has been amply demonstrated, and it is particularly called for with such reagents as ammonium oxalate and microcosmic salt.

## PART II.—METHODS APPLICABLE TO SILICATE ROCKS.

### 1. INTRODUCTORY REMARKS.

The order hereinafter followed in describing the various chemical separations has little relation to the affinities of the constituents of the rock, but those are grouped together which can be conveniently determined in the same portion of rock powder. Thus, in the main portion are usually determined  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{MnO}$ ,  $\text{NiO}$ ,  $\text{CaO}$ ,  $\text{SrO}$ ,  $\text{MgO}$ , total iron, and the combined weight of all the following:  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{ZrO}_2$ , all iron as  $\text{Fe}_2\text{O}_3$ , and nearly if not quite all vanadium, as  $\text{V}_2\text{O}_5$ , also perhaps rare earths if present. In a separate portion is estimated  $\text{FeO}$ , and also the total iron, as well as  $\text{BaO}$ , if these last are desired as checks. The alkalis need a portion for themselves. In another,  $\text{ZrO}_2$ ,  $\text{BaO}$ , rare earths, and total sulphur are very conveniently determined. For  $\text{V}_2\text{O}_5$  and  $\text{Cr}_2\text{O}_3$  still another and usually much larger portion is to be used. Determinations of  $\text{CO}_2$ ,  $\text{C}$ ,  $\text{H}_2\text{O}$ ,  $\text{F}$ ,  $\text{Cl}$  are all best made in separate portions of substance, the various combinations are possible, as  $\text{CO}_2$  and  $\text{H}_2\text{O}$ ,  $\text{C}$  and  $\text{H}_2\text{O}$ , or  $\text{H}_2\text{O}$ ,  $\text{F}$ , and  $\text{Cl}$ . In fact, by a judicious selection and combination of methods a very satisfactory analysis can sometimes be made on 4 grams of material without omission of anything of importance, though the time consumed will be greater than if ample material is available.

As an illustration of the advantage to be gained by a little judgment in the combination of methods, the case of sulphur, barium, zirconium, and rare earths may serve. Many chemists never look for any but the first of these, but by following the procedure given under A, page 115, not much more labor is expended in confirming their presence or absence than that of sulphur alone.

With only occasional exceptions nearly all the constituents mentioned on page 21 can be determined if present in portions of powder not exceeding 1 gram each in weight.

This is a convenient weight to take for the main portion in which silica, alumina, etc., the alkaline earths, and magnesia are to be sought; but it should, in general, be a maximum, because if larger the precipitate of alumina, etc., is apt to be unwieldy. Its weight can not often be much reduced with safety if satisfactory determinations of manganese, nickel, and strontium are to be expected. For the alkali

portion one-half gram is a very convenient weight. In general, it may be made a rule not to use more than 2 grams for any portion which has to be fused with an alkali carbonate, as for sulphur, fluorine, and chlorine. For carbon dioxide the weight may rise to 5 grams, or even more, if the amount of this constituent is very small, without expenditure of any more time than is required by 1 gram and with correspondingly greater approach to correctness in the result. For vanadium also a larger weight than 2 grams is usually demanded.

For the attainment of accurate results such as are needed for the close classification of a rock in the system devised by Cross, Iddings, Pirsson, and Washington,<sup>a</sup> too great stress can not be laid on the exercise of the utmost care throughout the analysis. This applies not only to routine manipulations, but even more in the matter of double precipitations, where these are called for, and in the testing of filtrates and precipitates to insure complete recovery of the desired substances, on the one hand, and freedom of them when obtained from matter which should not to be there, on the other. These cautions are especially urgent in the case of those having little experience. It must always be remembered that analysis can at best afford but approximate results, which will be the more serviceable the greater the care bestowed in obtaining them.

## 2. SPECIFIC GRAVITY. (POROSITY.)

### GENERAL REMARKS.

Though a knowledge of the specific gravity of rocks is seemingly less prized now than formerly by petrographers, it is occasionally needed and is ever a most important criterion in the identification of mineral species. As ordinarily carried out, whether by the method of suspension or by the pycnometer, the results fall short of the accuracy which is generally credited to them. A really accurate determination of the specific gravity of solids by the pycnometer, in which the error shall not exceed one or two points in the third decimal, is to be achieved only with the most painstaking precautions (see B. b. p. 43). Without this care the error in the second decimal may, and often does, amount to several points. For these reasons the subject of specific gravity is treated in some fullness. Although the porosity of a rock is a constant seldom used by the petrographer, it is one that is most useful in the comparative study of building stones, and since its determination as often carried out is likewise faulty and gives a value which does not represent the pore space in terms of the volume of stone, it has been deemed expedient to devote space to this subject also, especially as the determination of porosity involves that of specific gravity.

<sup>a</sup> Quantitative Classification of Igneous Rocks, 1903, University of Chicago Press, 286 pp.

## A. BY SUSPENSION IN WATER.

## a. ORDINARY METHOD FOR SPECIFIC GRAVITY.

This determination, when required, is best made on one or several fragments weighing up to 20 grams. They are held together by a fine platinum wire ready for suspension from the balance, and thus held are placed in a small beaker to soak over night in distilled water under the exhausted receiver of an air pump side by side with a similar beaker of water. Boiling is, of course, a much less effective means of removing air than the air pump, and the boiling water may exert an undesirable solvent and abrading effect. In the morning the wire is attached to the balance arm, the rock fragments remaining immersed in the water; a thermometer is placed in the companion beaker of water, now likewise in the balance case, and the weight is at once taken. Both vessels of water having precisely the same temperature, it is quite unnecessary to wait for the water to assume that of the balance should it not already possess it. The fragments are now lifted out, without touching the vessel, and carefully transferred to a tared crucible or dish. The wire is removed and at once reweighed, with the precaution that it dips just as far into the water now as when weighted. Hereby a special weighing of the wire out of water is avoided. The sample may now be dried on the water bath and then at 110° for some hours to certainly expel all absorbed water, and weighed after prolonged cooling in the desiccator. It is better to ascertain the weight of the dry rock after soaking in water than before, in order to avoid the error due to possible breaking off of a few grains between the two weighings. Should the density of the rock in air-dry condition be required, it may be left exposed to the air for a long period after drying and before weighing;<sup>a</sup> but the difference will only

<sup>a</sup>In view of the uncertainty as to what constitutes hygroscopic water (see c, p. 53), this course is perhaps more to be commended than the former, and seems imperative for certain zeolitic rocks. In such cases it is best to weigh the fragments before putting to soak, and afterwards to collect on a Gooch crucible the grains which may have fallen off in the water. Should no crucible of this kind be available, a paper filter may unhesitatingly be used and incinerated with the powder, owing to the small amount of which the error due to loss of even all its water during ignition is quite negligible.

Since the first publication of the foregoing in Bulletin No. 148, a series of determinations on eight rocks, in which the weighing was done after drying, led to an interesting observation. The lumps were heated for a short time on the steam bath until dry on the surface, then allowed to cool in air and weighed from time to time during two or three days to constant weight. It was observed that some lost weight from the start, while others gained. For instance:

14.3803	9.5342	17.0846
.3730	.5178	.1089
.3666	.5121	.1159
.3483	.5024	.1413
.3481	.5016	.1388
.3450	.5016	.1379
.3457		

The losses in the first and second cases may reasonably be ascribed to the gradual evaporation of water absorbed in the pores, while the increase in the third case is doubtless indicative of the presence of zeolitic constituents, from which even brief exposure to the heat of the steam bath had sufficed to drive out a good deal of their loosely held water. Subsequent careful analysis confirmed this view for each rock that gained in weight was found to hold a relatively large amount of water removable at 105°, and the microscope revealed the presence of zeolitization in them and not in the others. Hence, this procedure may be sometimes of value as a test for the presence of zeolitic minerals in advance of or as confirmation of the microscopic diagnosis.

in exceptional cases affect the second decimal by more than a single unit. For instance, an undried rock of 2.775 specific gravity containing in the uncrushed state the high percentage of 0.3 hygroscopic moisture will have a density of 2.79 when dry; a rock of 2.982 specific gravity, undried, will have a density of 3.00 after removal of 0.3 per cent of moisture. The difference becomes greater as the density of the rock increases.

This method of ascertaining the specific gravity of rocks is certainly more convenient than, and for compact rocks is believed to be decidedly preferable to, that of the pycnometer, in which the fragments must be reduced to small size with consequent formation of more or less powder, which is subject to slight loss in the various manipulations. To exclude this powder and employ only small fragments would introduce a possible source of error, since it is likely to consist largely of the most easily abraded minerals and consequently not to have the average composition of the mass. By following the instructions given above, loss of material is absolutely avoided, a decided saving in time is effected, and considerable weights can be easily employed with consequent lower probable error in the results. To vesicular rocks, however, notably certain lavas, the above procedure is, of course, inapplicable, since it is probable that many of the interior pores would be inaccessible to penetrating water.

#### b. METHOD FOR SPECIFIC GRAVITY AND POROSITY COMBINED.

E. R. Buckley<sup>a</sup> has called deserved attention to the faultiness of the methods generally employed for the determinations of these constants as applied to building stones, and has brought together in the work cited the fruits of his experience. His recommendations are essentially as follows:

The specimens should be cubes, such as are commonly employed for the other physical tests, measuring usually 2 inches square. These are dried for at least twenty-four hours at 110° (100° is not high enough to insure the removal of all interstitial water) and weighed to centigrams. They are then placed on the bottom of a large bottle, the cork of which can be hermetically sealed. Through the cork pass three tubes, two of which end below the cork and connect with a vacuum pump and manometer, respectively. The third tube, provided with a stopcock, ends near the bottom of the bottle and leads into a basin of water which can be heated to boiling. The bottle itself rests in a water bath, so that its contents can be kept hot all the time. At first the cubes rest in a shallow layer of water only, better to facilitate the escape of air and entrance of water under reduced pressure, which should not exceed one-twelfth of an atmosphere. The cubes should remain in this state for seventy-two hours,

<sup>a</sup> Building and ornamental stones of Wisconsin: Bull. Wisconsin Geol. Nat. Hist. Survey No. 4, 1888, pp. 63, 70.

experiment having shown that little or no absorption takes place after this lapse of time. Water is slowly let in from time to time, till the cubes are wholly immersed. In this way it is thought that thorough saturation can be attained. The saturated samples are transferred to an open dish filled with water and taken to the balance, where their weight in water is ascertained by suspension. They are then removed from the water, rapidly dried on the surface with bibulous paper, and the weights then taken in air.<sup>a</sup> They are dried at 110° for twenty-four hours and again weighed. The second dry weight was found by Doctor Buckley to vary rarely from the first by as much as 0.2 gram, and often by not more than 0.01. "When reckoned in per cent, the error would be such a small fraction of 1 per cent as not to be worthy of attention. The specific gravity was determined by dividing the average of the two dry weights by the difference between the dry weight and the weight of the cube suspended in water." For arriving at the true porosity or actual pore space compared with the volume of the sample tested, the following formula is to be used:

$$\frac{(B - A) \text{ sp. gr. } 100}{(B - A) \text{ sp. gr.} + A} = P$$

in which A is the average dry weight, B the saturated weight, and P the percentage of porosity or actual pore space. The value thus obtained is always much higher than that ordinarily taken as the measure of porosity. This last would better be called, according to Doctor Buckley, "ratio of absorption," being found by dividing the weight of water absorbed by the dry weight of the stone and multiplying by 100. Doctor Buckley finds no definite relation between porosity and ratio of absorption, although in a general way the former is a little more than twice the latter.

In calculating the weight of a stone in pounds per cubic foot the pores should be included in this volume. The weight sought is found by multiplying the apparent specific gravity by 62.5—that is, by the weight of a cubic foot of water. The apparent specific gravity can be calculated from the above data by the formula

$$\frac{A}{B - C}$$

in which A is the dry weight, B the saturated weight, and C the weight in water.

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<sup>a</sup> The above procedure differs from that of Doctor Buckley in the inversion of the order of weighing in water and after wiping. The more perfect the vacuum the less need be the temperature of the bath of immersion to insure the effective aid, by boiling of the water drawn up into the pores of the specimen, in securing expulsion of the inclosed air. A vacuum desiccator can perhaps be substituted for the bottle with much advantage. It will not be necessary to open the desiccator for the periodic introduction of water if to the evacuating tube is attached a Y or T with stopcocks, one branch leading to the pump and manometer, the other to a basin of water. By proper regulation of the cocks water can be drawn in at pleasure. In a later publication (Missouri Bureau Geol. and Mines, 2d ser., vol. 2, 1904) Doctor Buckley states that he has reduced the time of immersion to twenty-four hours and the vacuum to one-fourth of an atmosphere, without finding appreciable differences in the results obtained.

T. W. Thörner<sup>a</sup> and H. Seger and E. Cramer<sup>b</sup> have described apparatus alike in principle for determining porosity by the use of which a direct specific-gravity determination is avoided and the operations are very simple. The apparatus of Seger and Cramer are of simpler construction than those of Thörner and have stood the test of many years' use. By both, however, the true porosity as well as the "ratio of absorption" is ascertainable.

#### C. PENFIELD'S METHOD FOR MINERAL FRAGMENTS.

Penfield<sup>c</sup> recommends the following modification of the suspension method as more convenient than that by the pycnometer in many cases for small fragments of minerals.

After boiling in water, the substance is transferred with water to a small glass tube about 8 by 35 mm., provided with a fine platinum wire for suspension. This is weighed full of water in another vessel of water, and again after the removal of the mineral, the weight of which is found after drying.

This method is, of course, more applicable to homogeneous minerals than to rock fragments, and will therefore be applied in rock analysis chiefly to the determination of the specific gravity of the mineral grains separated by heavy solutions or acids.

#### B. PYCNOMETER METHOD.

##### A. ORDINARY METHOD.

If the pycnometer has to be used, as is generally the case when the density of any one of the mineral ingredients of a rock is desired after separation by one of the approved methods, it being then in a more or less finely divided state, a good procedure is that adopted in this laboratory by L. G. Eakins a number of years ago, provided there is no evaporation whatsoever around the stopper, a condition seldom, if ever, fulfilled, unless a thin film of vaseline is used, the weight of which need not exceed a half milligram. The pycnometer used is one with a capillary stopper, provided with a millimeter scale etched in the glass, the divisions being numbered both ways from the center and calibrated by mercury, so that the value of each one in weight of water is known. The capacity of the flask filled with water to the zero division is then calculated for every half degree of temperature from 0° to 30° by making a series of careful weighings in which, the capacity of the stem being known, it is quite immaterial at what level the water stands, provided it is within the limits of the scale. The exact temperature is obtained by an accurate thermometer placed in a companion vessel of similar shape to the pycnometer and containing

<sup>a</sup> Chem. Zeit., vol. 29, 1905, p. 744.

<sup>b</sup> Idem, p. 884.

<sup>c</sup> Am. Jour. Sci., 3d ser., vol. 50, 1896, p. 448.

like amount of water, both being left in the balance case till its temperature has been nearly or quite assumed, as shown by a second thermometer. The weighing must of course be made before the thread of water has sunk beneath the lowest division, which it will do after a time, even though at first filling the bore to the top of the stopper; and the corrected weight full of water to the zero mark is found by adding or subtracting the needed amount, as shown by the height of the thread on the scale.

For each pycnometer in use, and these are of different sizes, is prepared a table showing its weight, the value of each scale division in grams of water, and the capacity of the flask at different temperatures, as indicated above. The preparation of such a series of flasks at one time saved in the end, for the weighing of the flask full of water each time a density determination is made is rendered superfluous. All that is necessary is to look up in the table the weight corresponding to the temperature.

The density of the previously weighed substance in this case is now determined in much the same way as in A. a, p. 39, after the unstoppered pycnometer containing it and nearly filled with water has stood with its companion vessel of water under the air pump the necessary length of time. The water needed to fill the flask is taken from its companion.

All who have used the pycnometer method for fine substances know the difficulty experienced in preventing a portion from being held at the surface, despite all attempts at making it sink. Hence it often happens that a very small portion runs out around the sides of the stopper on inserting it. If the flask rests in a small tared dish the grains thus forced out may be washed down into it and weighed after evaporation in order to get the correct weight of that in the flask; or, after weighing, the contents of the flask may be emptied into a tared dish and the water slowly evaporated off in order to get the weight of the mineral. Usually this way is less to be recommended than the other.

#### b. REFINED METHOD.

In their elaborate study of the thermal properties of the feldspars<sup>a</sup> Drs. A. L. Day and E. T. Allen found the ordinary methods of specific-gravity determination of powders by the pycnometer to be affected with grave errors. In the following is given their procedure, slightly modified since publication, for obviating them to such a degree that with 25 cm.<sup>3</sup> pycnometers and 5 to 10 grams of material of from 2.50 to 2.75 specific gravity the error from all causes should never be greater than one unit ( $\pm 1$ ) in the third decimal place.

<sup>a</sup> Pub. Carnegie Institution of Washington, No. 31; and in less detail in *Am. Jour. Sci.*, 4th ser., 1. 19, 1905, p. 93.



A chief source of error is the evaporation of water around the stopper. In order to reduce this to a negligible quantity, the grinding should be very fine and the stopper should be slightly vaselined before the final weighing. If the excess of vaseline is carefully wiped off, the error due to its weight will not affect the result in the third decimal with 25 cm.<sup>3</sup> flasks. The best flasks are those with capillary stoppers, and Day and Allen now have them made with necks 2 mm. thick, so as to avoid springing when the stopper is pressed in, and ground square off to prevent the adhesion of a film of water between stopper and neck above the joint. With the stopper slightly vaselined, such a flask does not lose at 30° more than 1 to 1½ milligrams per half hour.

To obviate another serious source of error, incomplete removal of air from the powder and undue loss of powder during filling and stop-

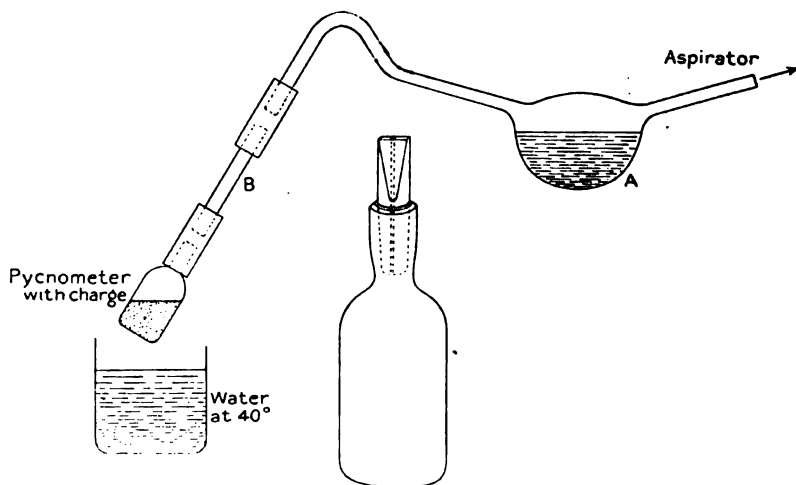


FIG. 7.- Moore's device to remove air from mineral powders.

pering, the device of G. E. Moore<sup>a</sup> is made use of, slightly modified, as shown in fig. 7. The flask containing the powder is attached to a water pump, with the intervention of a tube and bulb attachment, as shown. The bulb A contains boiled water. When the system has been nearly freed from air by the pump, water from A is made to flow into the flask. Then by tapping and plunging the latter into water warmed to 40° to 50°, in order to produce boiling within the flask, the air is soon removed. During the boiling powder will usually be thrown up into the connecting tube; but this can be washed back by water from A or, after disconnecting, by boiled water from a wash bottle. Should any particles remain in the tube, they are washed into a tared dish and weighed, after evaporation of the water. It is absolutely essential that not the smallest grain be caught between the

<sup>a</sup> *Am. Jour. Sci.*, 3d ser., vol. 3, 1872, p. 41; *Jour. prakt. Chemie.*, vol. 2, 1870, p. 329.

stopper and the neck. To this end the neck is wiped out with a bit of filter paper, which is burned in the tared dish in order to obtain the proper correction.

Constant temperature is attained in a water thermostat electrically regulated to tenths of a degree at a temperature slightly above that of the balance case. In the thermostat there is also placed a small vessel with water for the final filling. If the powder is very fine, the flask may have to remain in the thermostat several hours to allow complete settling before wiping the neck, vaselining, and stoppering. When filled and stoppered, it is advisable to return it to the thermostat for a time, with a short piece of rubber tube slipped part way over the end of the stopper, so as to form a cup for a drop of water and insure that the capillary remains filled. At the proper time the rubber is removed and the flask quickly wiped with bibulous paper and weighed without delay. A blank test must of course be made with water alone, or the pycnometer may be calibrated once for all within any desired range of temperature. A determination of the accuracy attainable in this way is subject to a correction for buoyancy.

(See p. 55 for remarks by Doctors Day and Allen on another source of error in the determination of the specific gravity of powders.)

#### C. HEAVY SOLUTIONS NOT SUITABLE FOR ROCKS.

Because of their roughness, porosity, and complex mineral composition the density of rock fragments can not be accurately determined by that of heavy solutions in which they may remain suspended.

### 3. PREPARATION OF SAMPLE FOR ANALYSIS.

#### A. QUANTITY OF ROCK TO BE CRUSHED.

In the great majority of cases a few chips from a hand specimen will well represent the average of the mass, but with rocks in which a porphyritic structure is strongly developed the case is different. Here a large sample should be provided, gaged according to the size of the crystals, and the whole of this should be crushed and quartered down for the final sample. Unless this is done, it is manifest that the analysis may represent anything but the true average composition of the rock.

#### B. CRUSHING.

For accurate analyses the use of steel power crushers and mortars is out of the question because of the danger of contamination by particles of metal and the impossibility of cleansing the roughened surfaces after they have been in use a short time. Extraction, by the

aid of a magnet, of steel particles thus introduced into the powder is quite inadmissible, since the rocks themselves, almost without exception, contain magnetic minerals.

#### A. SURVEY METHODS.

For breaking large pieces of rock to small sizes a thick iron plate with specially hardened surface and a similarly hardened pounder, such as street pavers use, will probably render the best service, but the hardening must be done with extreme care.

A method of rough crushing on a small scale that gives fair satisfaction in practice is to place each fragment as received on a hard steel plate about  $4\frac{1}{2}$  cm. thick and 10 cm. square, on which is likewise placed a steel ring 2 cm. high and of about 6 cm. inner diameter, to prevent undue flying of fragments when broken by a hardened hammer. In this way a considerable sample can soon be sufficiently reduced for transfer to the agate grinding mortar with a minimum of metallic contamination.

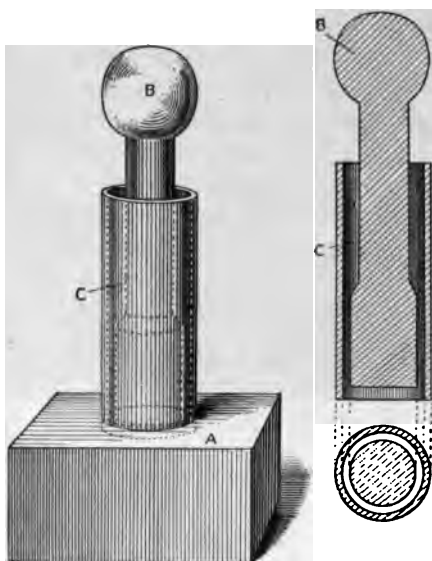


FIG. 8.—Ellis's mortar for crushing coarse materials.

All parts of best chilled and surface-hardened tool steel. A, Block,  $12\frac{1}{2}$  by  $12\frac{1}{2}$  by 6 cm., with depression in center 0.6 cm. deep; B, pestle, 20 cm. high, 3.5 cm. diameter at base; C, cylinder,  $12\frac{1}{2}$  cm. high, 5 cm. outside diameter, 4 cm. inside diameter, fitting accurately the depression in the block.

The cylinder fits snugly into a depression in the block, after the manner of an ordinary "diamond" mortar, but the pestle has a diameter less than the inner diameter of the cylinder. The crushing is done by the pestle, without the aid of a hammer.

An imperative precaution, when using either of these methods, however, is to refrain absolutely from the least grinding or rubbing motion, with the hammer in the one case or the pestle in the other. Even quartz can be broken down by vertical blows to a size fit for the agate mortar without showing any darkening in color, whereas if a rubbing motion has been employed to even a limited extent, the

der will, by contrast, be perceptibly dark after grinding in the mortar. The hardest steel is very susceptible to abrasion by minerals harder than quartz. The crushing must be carried to such a degree of fineness that after transferring the grains to the mortar the sliding motion of its pestle will not occasion further loss of flying particles, since the portions lost will, in case of all but glassy or very grained material, not have the same composition as those retained.

#### B. WASHINGTON'S METHOD.

The rock is first reduced, as by the methods in A, to lumps that will fit into a "diamond" steel mortar of Plattner's form, the bottom of the cavity being hemispherical and matched in shape by the end of the pestle. The lumps are crushed, one by one, in the mortar by a series of blows of a half-pound hammer (billet of wood, Dittrich), and the contents of the mortar are emptied into a cylindrical glass box 3.5 cm. deep and 7.5 cm. internal diameter, with walls about 2 mm. thick. When the whole sample, or a quartered portion if large, has been thus crushed a piece of best silk bolting cloth with about 10 meshes to the centimeter is stretched over the open side of the box and clamped fast by a brass ring 1 cm. in height. The box is then inverted and the finest powder gently sifted onto a sheet of clean paper. The material left on the cloth is recrushed in the mortar and resifted, these operations being repeated until all has passed the sieve. It will, of course, not do to reject any portion, for thereby the composition of the mass will be affected. The sample is now fine enough to be reduced by grinding in a large agate mortar.

Doctor Washington finds that in this way there is practically no danger of contamination by metallic iron, and that any error resulting from introduction of cloth fiber is negligible. This last hardly can be true of the method practiced in parts of Europe (the German "beuteln"), which differs in essential points from the gentle method of Doctor Washington.

#### C. GRINDING.

Ordinarily an extremely fine state of division is unnecessary, except in the case of those portions in which alkalies and ferrous iron are to be determined, or where soluble constituents are to be removed by acids, etc., and in such cases the final grinding can be done at the balance table on a small portion slightly in excess of the quantity to be weighed off.

Formerly all rock samples in the Survey laboratory were, after weighing, reduced to powder by hand grinding, with expenditure of much time and labor. Since then, except in special cases, the work has been done by mechanical grinders run by a small electric motor. The grinder manufactured by the McKenna Brothers Brass Com-

pany, Limited, of Pittsburg, Pa., is shown in fig. 9, with the addition of simple attachments intended to keep metal dust and oil out of the mortar. These additions have been found to be quite essential for our work. As made for the technical industries the machine is



FIG. 9. - McKenna ore grinder, slightly modified by the addition of the following attachments to prevent oil and dust from belts and bearings from entering the mortar. A, Copper cup soldered to head of pestle holder. This partakes, without the slightest friction of the motion of the latter. B, Tin plate with deep sinus, the latter being covered in turn by the rubber cloth, C, which fits closely about the metal shaft. The spring at the top of the sliding rod, to which the agate pestle is attached at the bottom, can be adjusted to give any desired pressure or can be thrown back entirely to allow the pestle to be raised in removing the agate mortar. This is accomplished by loosening a set screw and dropping one of the four posts holding the mortar in place. The pestle should make about 200 revolutions per minute, the mortar moving very slowly in the same direction. The scraper keeps the ore in the center of the mortar and the combined rolling and sliding motion controlled by the ball and socket supporting side arm reduces the hardest ore very rapidly." - *Iron Age*.

largely of brass, nickel plated, but the latest made for the Survey is of steel throughout, without plating. It was found that the pulley belts caused too much abrasion of the softer metal to permit its use for much of our work, on account of the danger of copper, zinc, and

nickel getting into the sample in traces, and that in spite of a very limited use of oil this invariably became scattered over the contents of the mortar, though its presence never became visible to the eye except when white materials were ground. The last contamination is prevented by the copper cylinder, A, soldered securely onto the pestle holder so as to form a cup large enough for the purpose intended. The tin shield, B, with a superposed rubber cloth, C, covering the slit required for the proper adjustment of the shield, greatly lessens the danger of metallic contamination, even with the all-steel machine. At first it was thought that a rubber cloth covering the mortar, with the pestle snugly fitting a hole in the cloth, would be an effective protection, but it was found that there was sufficient friction on the rubber itself to cause contamination of the powder beneath.

With this apparatus a large amount of work can be done in a short space of time, though there is much loss of fine powder if the speed of revolution is too high. In our actual practice, three of these machines are operated, one or all, on a single table from the same shaft, the whole being covered with a glass case, divided by glass partitions, so as to exclude dust and separate the grinders.

The abrasion of pestle and mortar becomes far more manifest with these machines than with hand grinding, notwithstanding that each has its motion simulating as far as possible that of the hands. There is undoubtedly a very considerable introduction of silica into the sample from these sources, but in comparison with the amount invariably present in an igneous rock the quantity abraded is hardly of moment. Rocks normally free from silica, or nearly so, like many iron ores, are as a rule softer than igneous rocks and consequently cause less abrasion of the grinding implements.

W. Hempel<sup>a</sup> has experimentally tested the effect of grinding in mortars of agate, glass, iron, and hardened steel, and has found that for hard bodies, like glass, hardened steel is far superior to agate, and that even mortars and pestles of green bottle glass showed much less abrasion than agate. In grinding 10 grams of glass to very fine powder the agate mortar and pestle, weighing 416 grams, lost 0.052 gram in weight (or 5 milligrams per gram of glass), against a tenth of that loss with a hardened steel mortar and pestle of the same weight that had been some time in use.

Notwithstanding these observations in favor of steel, the use of metal in rock analysis is, as said above, forbidden by the conditions of the case. Still more so is that of glass because of its complex composition. If impurity can not be avoided it is far better that it should be of one kind only, and that one productive of less disturbance than any other.

Metal sieves should never be used.

<sup>a</sup> *Zeitschr. angew. Chemie*, 1901, p. 843.

## D. WEIGHT OF GROUND SAMPLE.

The sample when ground should weigh not less than 10 grams, and preferably 20 in case it should be necessary to repeat or advisable to employ unusually large portions for certain determinations, notably carbonic acid. Rock analysis has in this respect an advantage over mineral analysis, since material is almost always available in ample quantity and any desired number of separate portions may be used, whereas with a mineral the analyst is frequently compelled to determine many or all constituents in a single, often very small, portion of the powder. This course often involves delay and the employment of more complicated methods of separation than are usually necessary in rock analysis.

## 4. WATER—HYGROSCOPIC, ZEOLITIC, CRYSTAL.

## A. GENERAL CONSIDERATIONS.

## a. IMPORTANCE OF EMPLOYING AIR-DRY POWDER FOR ANALYSIS.

The time-honored custom of drying a powdered specimen before bottling and weighing has long seemed to me one that has no sound basis in reason. Its object is of course plain, namely, that of securing a uniform hygroscopic condition as a basis for convenient comparison of analytical results, since some rocks contain more hygroscopic moisture than others. Nothing, however, is more certain than that by the time the substance is weighed it has reabsorbed a certain amount of moisture, small indeed in most cases, but very appreciable in others; and further, with every opening of the tube moisture-laden air enters and is inclosed with the remainder of the dry powder. It therefore may very well happen that a powder at first dry will, after several openings of the tube, especially at considerable intervals, be nearly as moist as when first inclosed. Doctor Dittrich has expressed views similar to the above.

It is preferable to weigh the air-dry powder and to make a special determination of moisture. If all the portions necessary for an analysis are weighed out one after another, or even at different times on the same day, the error due to difference of hygroscopicity in dry and moist weather, which for most of the separate portions is an entirely negligible quantity, is eliminated. Only in the main portion, in which silica and the majority of the bases are to be estimated, can it ever be an appreciable factor.

## b. TEMPERATURE OF DRYING.

As to the temperature to be adopted for drying in order to determine so-called hygroscopic moisture, the practice has varied at different times and with different workers, ranging from 100° to 110°. For the great majority of rock specimens it is quite immaterial which of

these temperatures is adopted, since no greater loss is experienced at the higher than at the lower temperature, given a sufficient time for the latter. It is the present practice in this laboratory to employ a luene bath giving a temperature of about  $105^{\circ}$ . Should the results show a very unusually high loss, the powder is reheated at, say,  $125^{\circ}$ , in order to learn if the loss is progressive with increased temperature. In the affirmative case it may be well to repeat the drying at  $100^{\circ}$ , for a portion of the loss at  $105^{\circ}$  was probably due to combined water from a mineral or minerals in the rock; but in that case even the loss at  $100^{\circ}$  may sometimes very well include combined water, in which case drying over sulphuric acid alone may be desirable, or over dry sand.

## C. INDIRECT METHODS.

In the latter connection it is proper to point out certain pitfalls in the path of the unwary, which, however, are in part more likely to be counteracted in the analysis of minerals, where their influence may be of far-reaching consequence.

A mineral which loses a great deal of water over sulphuric acid—2 or 3 per cent, for instance—may need an exposure of several days or even weeks for its complete extraction. If the weighings are made from day to day, the apparent limit may be reached long before all water actually removable has been taken up by the acid. Whenever the crucible, after weighing, is replaced in the desiccator it is no longer in a dry but a more or less moist atmosphere, and its contents, even when covered, sometimes absorb a part of this moisture and retain it so persistently that the acid is unable to bring the powder beyond its previous state of dryness in the next twenty-four hours. In fact, it may be unable even to reach it unless greater time is allowed. An experiment on 1 gram of tyrolite, made and published some years ago, seems to illustrate this point in part:

*Experiment of drying 1 gram of tyrolite.*

Time exposed.	Loss.	Time exposed.	Loss.
<i>Hours.</i>	<i>Gram.</i>	<i>Hours.</i>	<i>Gram.</i>
18	0.0231	24	0.0002
26	.0083	24	.0003
23	.0029	48	.0006
24	.0012	24	.0002
23	.0008		
24	.0001	283	.0380
25	.0003		

The experiment might reasonably have been considered ended after one hundred and fifty-eighth hour, when a loss of but 0.1 milligram was shown during twenty-four hours; but nevertheless a nearly steady loss of 0.3 milligram per day took place for six days more, and might have been longer observed but for the interruption of the experiment.



Again, it is a common practice to determine the water given off by hydrous minerals in an air bath at temperatures at and above  $100^{\circ}$ . To insure accuracy this experiment should never be made in crucibles or dishes which must be cooled in a desiccator. One instance will suffice: A gram of a mineral mixture containing about 17 per cent of water, of which about 3 per cent was driven off at  $100^{\circ}$  and 8 or 9 per cent at  $280^{\circ}$ , was, after several hours' heating at the latter temperature placed in a desiccator over sulphuric acid and weighed as soon as cold, then replaced and again weighed the next day. It had regained  $1\frac{1}{2}$  per cent of its original weight, although the desiccator was tightly closed and the crucible covered, showing apparently a drying power superior to that of the acid.

A specimen of tyrolite was found on one occasion to lose 10.34 per cent at  $280^{\circ}$ , and on another occasion 14.33 per cent. In the latter case the drying and heating at progressive temperatures had continued during a period of 528 hours, the weighings being made usually from day to day; whereas in the former the duration of the experiment was much shorter and the intervals between weighings were but a few hours each.

An important research of C. Friedel<sup>a</sup> well shows what errors are possible in the determination of this easily removable water, since he found that certain zeolites which had been largely dehydrated but not heated to the point of rupture of the molecular net could then absorb, instead of water, various dry gases in which they might be placed, as carbon dioxide, ammonia, carbon disulphide, and others, even air in large quantities, and certain liquids. In the light of this observation the cause of the great increase of  $1\frac{1}{2}$  per cent in weight of the partially dehydrated mineral mentioned above may very possibly be attributed to air from the desiccator instead of moisture, as was at the time supposed. At any rate, as Friedel says, the danger of accepting a loss in weight as an index of the amount of water lost is clearly shown, and thus that method of determining water is for many cases fully discredited. Just what method to adopt must be largely left to the judgment of the operator, who will often be guided by the mineral composition of the rock as revealed by the unaided eye or the microscope.

Friedel<sup>b</sup> indicates a means for determining the true weight of water lost by minerals behaving like the zeolites, even without collecting the water lost, namely, by driving out of the dehydrated and weighing mineral, under proper precautions, any air it may have absorbed in the process of drying and cooling, and collecting and measuring the air and thus finding its weight, which, added to the apparent loss, gives the true contents in water.

<sup>a</sup> Bull. Soc. Min., vol. 19, 1896, pp. 11, 91; Comptes rendus, vol. 122, 1896, p. 1006.

<sup>b</sup> Loc. cit.

I have shown<sup>a</sup> the importance of employing in desiccators only sulphuric acid of maximum dehydrating power. Acid that has stood long in desiccators that are in constant use is by no means so efficient as fresh, strong acid.

But aside from the above reasons for avoiding the use of indirect methods of water determination, there is another very important one. They never give correct results where air baths are used in the ordinary way, for the reason that the drying is performed in an atmosphere far from dry, and the substance can not, therefore, lose all its moisture. As examples, if the indirect method shows 0.12 and 0.75 per cent in two samples, the direct method may afford 0.17 and 1.00 per cent respectively. Similar observations have been repeated many times, and admit of no dispute. They apply as well to rocks as to minerals. Therefore, if the moisture content is more than an insignificant factor the indirect method must be avoided, or so arranged as to insure a constantly changing dry atmosphere.

To effect this the powder should be heated in a weighed tube, through which a current of dry air can be passed, and allowed to cool therein, after which a second weighing gives the loss of moisture. The degree of drying will depend on the nature of the reagent used for drying the air, sulphuric acid being more effective than calcium chloride, and phosphoric pentoxide most effective of all. When using the last, it is well to cause the air first to pass over or through one of the first named dehydrants, for its effectiveness rapidly lessens as it becomes glazed with metaphosphoric acid. For experiments carried out at room temperatures no bath is required, but for artificial temperatures the tube should pass through a suitable thermostat

#### d. DIRECT METHOD.

In general it is preferable to collect and weigh the moisture in suitable absorption tubes after its expulsion by the aid of apparatus described in B, page 56, even though the long time often required is sometimes an objection, since the absorption tube may gain weight, heavier than that of water from the mineral, sufficient to introduce an appreciable error. It may in such cases be necessary to run a long blank test for the correction to be applied.

#### e. ARGUMENT IN FAVOR OF INCLUDING HYGROSCOPIC WATER IN SUMMATION.

The question has been asked: "If the so-called hygroscopic water is not always such, but not infrequently includes combined water, why not its determination and separate entry in the analysis entirely unnecessary? Why make a distinction, which, after all, may not be

<sup>a</sup> Am. Chem. Jour., vol. 14, 1892, pp. 6-7.

a true one?" The question involves the further consideration of the advisability of including in the analysis at all the loss at 100° or 110°.

It would seem to be a reasonable desire on the part of petrographers to have all analyses referred to a moisture-free basis in order that they shall be strictly comparable. This involves the omission of the "hygroscopic" water from the list of constituents, which would be eminently proper were it always possible to be sure that the loss at 100° to 110° truly represents mechanically held water. Since it very often represents more, and the determination as to whether or not it does in each case is not always possible and would add to the time required for the analysis, it seems necessary to include this water. What errors may arise from its exclusion the following rather extreme case well illustrates: Certain rocks of Wyoming in powder form lost from 1 to 2 per cent of moisture at 110°. That not even an appreciable fraction of this was truly hygroscopic is fully demonstrated by the fact that the uncrushed rocks lost the same amount; yet the rule followed by many chemists and petrographers would have involved the removal of all this water as a preliminary to beginning the analysis, and not only would a most important characteristic have passed unnoticed, but the analyst would have reported an incorrect analysis, inviting to false conclusions and possibly serious confusion. (See also second paragraph of footnote, p. 39.)

f. SEPARATE ENTRY OF HYGROSCOPIC AND COMBINED WATER.

To revert now to the primary question, it may be said that the estimation of the loss at 100° or 110° and its separate entry in the analysis is advisable as not infrequently affording at once to the lithologist an indication of the mineral character of one or more of the rock constituents, thus perhaps confirming the microscopical evidence or suggesting further examination in that line. An unusually high loss at 100° would be regarded as probable evidence of the presence of zeolites or other minerals carrying loosely combined water. It has been objected that the true hygroscopic moisture varies with the degree of comminution of the sample and with the condition of the air at the time of weighing, and that it is therefore improper to incorporate it in the analysis; but this variation is ordinarily not at all great. Perhaps the time may come when it will be the rule to ascertain by additional heating at a higher temperature whether the water lost at 100° is to be regarded as purely hygroscopic. In such case it would be proper to omit it, and a distinct advance would undoubtedly be scored.

g. IS ALL TRUE HYGROSCOPIC WATER EXPELLED AT 100°?

It has been tacitly assumed in the foregoing that true hygroscopic water can all be expelled at 100°, which perhaps is not to be accepted as *universally true*. Eminent authority holds that it is impossible,

in the cases of certain foliaceous minerals, notably the micas, to thus entirely remove it, and that a part is driven off only at higher temperatures. If this is true, a further uncertainty is introduced in its determination, which not only strengthens the argument in favor of entering all water in the tabulation, but also serves to emphasize the difficulties of the situation.

The following excerpt from the work of Doctors Day and Allen<sup>a</sup> on the feldspars, already cited, has direct and important bearing on the above.

There is another error to which accurate specific-gravity determinations upon powdered minerals will be subject unless suitable precaution is taken. The exposure to the air during the period of grinding the samples gives opportunity for the condensation of sufficient atmospheric moisture upon the grains to affect the weight in air. The amount varies measurably with the size of the grains, as will be seen from the accompanying data, and probably with the degree of saturation of the atmosphere and the time of exposure.

*Determination of moisture in 1 gram of powdered mineral upon exposure to the air.*

[<=finer than. >=coarser than.]

Mineral.	Fineness (mesh).	Moisture.
		<i>Gram.</i>
Orthoclase (natural glass).....	<150.....	0.0061
Ab <sub>1</sub> An <sub>3</sub> (artificial glass).....	Selected, coarse.....	.0000
Ab <sub>1</sub> An <sub>3</sub> (artificial crystal).....	<100 >120.....	.0010
Ab <sub>1</sub> An <sub>1</sub> (artificial glass).....	<100 >120.....	.0007
Ab <sub>1</sub> An <sub>1</sub> (artificial crystal).....	<100 >120.....	.0010
Ab (natural crystal).....	Coarse.....	.0006
Do.....	<150.....	.0069
Orthoclase (natural crystal).....	<120 >150.....	.0011
Orthoclase (same sample).....	<150.....	.0031
Do.....	Still finer.....	.0050
Orthoclase (artificial glass).....	Everything <100.....	.0065
Orthoclase (portion of same).....	>150.....	.0022

In the last two groups, note that the moisture in graded portions of the same sample varies with the fineness.

We also verified the conclusion of Bunsen<sup>b</sup> that this absorbed moisture is not altogether removed at temperatures only slightly above 100°, but requires 600° to 800°—equivalent to low red heat. Several samples for which the moisture had been determined were laid away in corked test tubes for a number of weeks, after which redetermination gave exactly the former value.

It is worth noting in this connection that these measured quantities of absorbed water are of the same order of magnitude as those usually obtained for the water content in feldspar analyses, where again, of course, the finer the sample is ground for the analysis the greater the possible error from this cause. It may be that a part and occasionally all of the moisture usually found in these analyses is adsorbed and the significance of its presence there mistaken.

<sup>a</sup> Pub. Carnegie Institution of Washington, No. 31, pp. 56-57; Am. Jour. Sci., 4th ser., vol. 19, 1905, p. 93.

<sup>b</sup> Wied. Ann., vol. 24, 1885, p. 327.

## B. APPARATUS FOR THE DIRECT DETERMINATION OF WATER AT DIFFERENT TEMPERATURES.

## GENERAL PRECAUTIONS.

A precaution too often overlooked is to see that the drying agent used for the air current is the same as that in the absorption tube in which the water from the sample is collected—not only the same in kind, but as near as may be in strength as well, especially with calcium chloride. Therefore, the contents of drying and absorption apparatus should be changed often, and when refilled the reagents should be taken from the same stock. Fresh calcium chloride in absorption tubes should be exposed to a current of carbon dioxide and the excess removed by air before use. Furthermore, all connections should be as nearly as possible glass against glass, with the least exposure of rubber. With long rubber connections the error may be a very sensible plus one. Again, between refillings of the absorption tube the direction of the air current through it should always be the same. Care must also be taken that the conditions at the several times of weighing of the tubes are nearly alike as to temperature and hygrometric state of the atmosphere, for the afternoon weight of a tube may be appreciably different from that on the following morning. Finally the electrifying effect of wiping a tube before placing it on the balance must not be overlooked. In my experience this has often amounted to as much as 1 to 2 centigrams (always an increase in weight) after wiping with a clean linen handkerchief. The charge can be removed by repeated application of the hand, but disappears slowly without such aid. A strongly electrified condition makes itself apparent by marked irregularities in the swing of the needle. While more pronounced in cold weather, the phenomenon is not peculiar to winter.

## B. STEIGER'S APPLICATION OF THE TOLUENE BATH.

The apparatus devised by George Steiger consists simply of a U-tube of home construction and a small toluene bath, in the cover of which is an opening of suitable shape to receive the tube (fig. 10). The open space between the uprights is closed when in operation by a piece of asbestos board. The horizontal end of the tube is made long, so that in the event of much water being given off it can be readily driven over into the absorption tube by a low flame, which if applied from the first may prevent any appreciable condensation of water before reaching the calcium chloride. The interior of the tube having been first freed from adhering moisture by a current of dry air and the absorption tube attached, the stopper is lifted without shutting off the

current, and the weighed powder quickly introduced by means of the metal trough in which it has been weighed (fig. 4, p. 31). The stopper is as quickly replaced and the dehydration begins at once and is often nearly finished by the time the thermometer indicates the maximum temperature.

By employing a U-tube with glass stoppers, like A, fig. 11, it would be quite possible to ascertain the loss in weight of the powder as well as the gain of the absorption, or to do away with the latter altogether. In this case, however, it would be necessary to seal the stoppers very tightly, and perhaps to reduce the powder through a dry funnel because of the vaseline film on the inner side of the end of the tube. The moderate heat on the top of the bath would suffice to prevent condensation of moisture before reaching the absorption tube, and to soon remove what might condense at first, but there must be no rubber connections. By employing a suitable oven this form of apparatus can be used for higher temperatures, but the stoppers must fit airtight, for vaseline can not be employed.

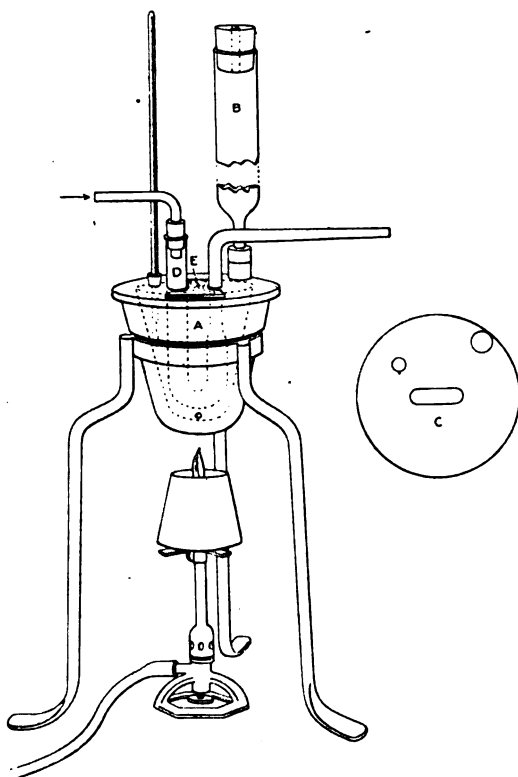


FIG. 10.—Steiger's form of drying apparatus. A, Toluene bath; B, condenser, about 50 cm. high; C, cover of bath, showing openings; D, tube for mineral powder; E, asbestos board cut out so as to close central opening in cover.

#### B. CHATARD'S OVEN FOR TEMPERATURES UP TO 300° AND OVER.

A form of drying oven devised by Dr. T. M. Chatard<sup>a</sup> is in use in this laboratory for determining water at different temperatures up to 100°, and gives entire satisfaction. It is an asbestos-covered copper box, B, shown in different aspects and parts in fig. 11. The box is constructed that the tube with its contents can be removed without detaching from either the drying or collecting tubes, which is a great advantage if it is desired to apply afterwards the direct heat

<sup>a</sup> Am. Chem. Jour., vol. 13, 1891, p. 110; Bull. U. S. Geol. Survey No. 78, 1891, p. 84.

of a lamp in order to expel the water retained at  $300^{\circ}$  to  $350^{\circ}$ . To facilitate this removal the stand is on rollers, so that after clamping the projecting end of the tube and removing the front of the box, F, and the little side pieces, S, closing the horizontal slits, the oven can be rolled bodily backward, leaving the tube and its attachments in their original position, ready for further heating over a burner or blast. The removable front, F, of the oven is made of two pieces of sheet asbestos board stiffened by an interlaid piece of sheet copper. The inner piece of asbestos board fits snugly into the box, while the outer

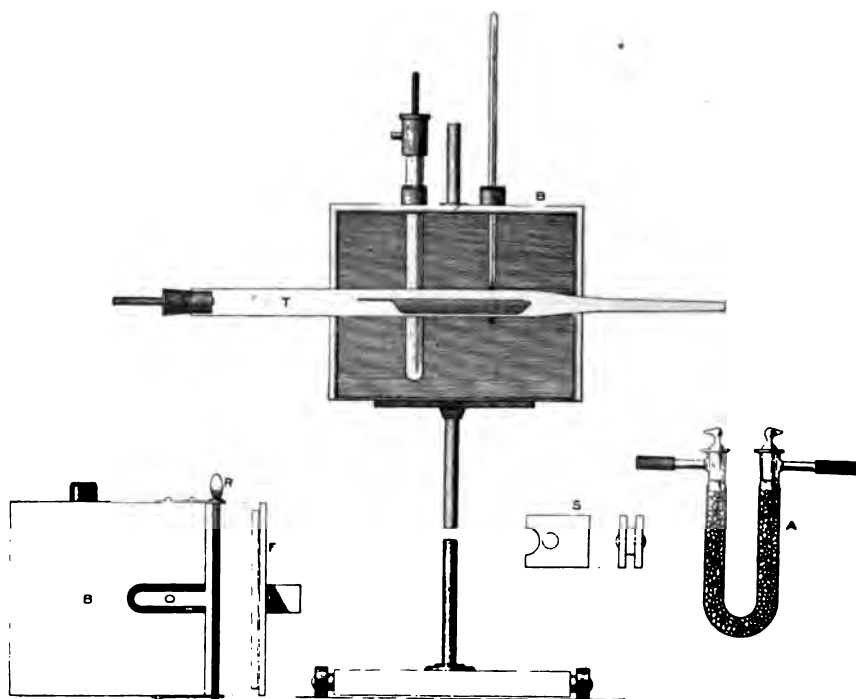


FIG. 11. — Chatard's form of drying oven for water determinations. B, Copper box, 18 cm. long, 10½ cm. high, 9 cm. wide, open in front, its sides and top covered with asbestos board; S, two slides of different sizes to close openings, O, after the tube is in position; F, asbestos-board front stiffened by an interlaid sheet of copper; R, metal rod to hold front in place; T, glass ignition tube; A, calcium-chloride absorption tube. For mineral substances with very high water content the tubes T and A may preferably have the original form given them by Chatard, as depicted in Bull. No. 176, p. 37. The use of a straight tube, as shown in fig. 11, permits of a less complicated construction of box and stand than that here shown, since round holes in opposite ends of the box admit the ignition tube, and the roller base is then superfluous.

one, being slightly larger, by its projecting edges hinders the door from falling in and helps to prevent air currents. This door is held in place by the metal rod, R. The little slides, S, are made in a somewhat similar manner, and are intended to slip in from the front and close the two openings, O, after the tube is in place, but before closing the front.

For other forms of tubes adapted to similar determinations, see pages 61 and 66-67.

## 5. WATER—TOTAL OR COMBINED.

## A. ARGUMENTS AGAINST "LOSS ON IGNITION" METHOD.

In a few cases the simple loss on ignition of a rock will give the total water with accuracy, but in the great majority there are so many possible sources of error that this old-time method can rarely be used with safety. Only when the rock is free from fluorine, chlorine, sulphur, carbon, carbon dioxide, and fixed oxidizable, or reducible ( $\text{MnO}_2$ ) constituents can the loss be accepted as the true index of the amount of water present, and it is rare that a rock is met with fulfilling these conditions, especially as to the absence of ferrous iron. Blast ignition in presence of carbon dioxide alone of the above list may give a correct result, after separate estimation of the carbon dioxide, provided this emanates from carbonates of the earths and not from those of iron or manganese. The long-maintained idea that in presence of ferrous iron a sufficiently correct result is obtainable by adding to the observed loss an amount needed for oxidizing all ferrous iron is not justifiable. There can be no certainty that the oxidation has been complete, especially in the case of readily fusible rocks, and at the high temperature of the blast a partial reduction of higher oxides is not only possible but sometimes certain. The inability to ensure complete oxidation by simple ignition is illustrated in the case of precipitated ferric hydroxide which has been ignited in contact with its filter paper. If the quantity was in any degree large it is sometimes decidedly magnetic, presumably from presence of magnetic oxide, which no amount of heating wholly oxidizes, especially in the larger grains. Neither is evaporation with nitric acid and re-ignition sufficient to destroy the magnetic property of the oxide, as has been claimed.

Direct weighing of the water evolved is then imperative in most cases, and of the numerous methods advocated, or in general use, several will now be considered.

## DIRECT WEIGHING OF THE WATER WITHOUT THE USE OF ABSORPTION TUBES—PENFIELD'S METHODS.

## a. FOR MINERALS EASILY DEPRIVED OF THEIR WATER.

If no other volatile constituents than water are present, the beautifully simple method first used by Prof. G. J. Brush and extended by Prof. S. L. Penfield<sup>a</sup> leaves nothing to be desired for accuracy. It consists simply in heating the powder in a narrow tube of hard glass, enlarged at the closed end and provided with one or two further enlargements in the middle to hold the water and prevent its running

<sup>a</sup> *Am. Jour. Sci.*, 3d ser., vol. 48, 1894, p. 31; *Zeitschr. anorg. Chemie*, vol. 7, 1894, p. 22.



back and cracking the hot glass. A capillary glass stopper fitted in with rubber tubing prevents loss of water by circulating air currents. The tube being held horizontally, the bulb is heated to any required degree by the Bunsen or blast flame. Moistened filter paper or cloth wound about the cooler parts of the tube insures condensation of all water. The heated end being finally pulled off, the tube is weighed after cooling and external cleansing, and again after the water has been removed by aspiration. For most rocks, as they contain little water, central enlargements of the tube are hardly needed.

Various forms of tubes used by Penfield are shown in fig. 12.

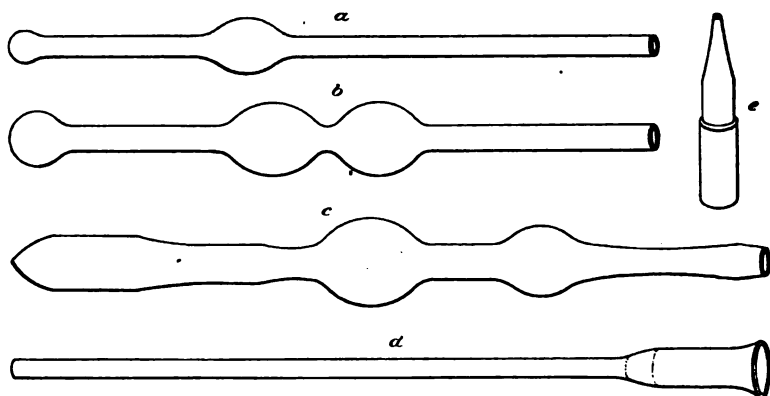


FIG. 12. --Penfield's tubes for water determination in minerals. *a*, *b*, *c*, different forms of tubes; *d*, thistle tube for introducing the powder; *e*, capillary-tipped stopper.

Before using, even if apparently dry, "these tubes must be thoroughly dried inside, which is best accomplished by heating and aspirating a current of air through them by means of a glass tube reaching to the bottom."

How this simple tube is made to afford entirely satisfactory results with minerals, even when carbonates are present, is fully set forth in the paper cited.

Few rocks, comparatively, are altogether free from other volatile constituents. Hence, for refined work the application of this apparatus in the simple manner above set forth is limited. It may, however, be used with the addition of a retainer for fluorine, sulphur, etc., in the shape of calcium, lead, or bismuth oxides.

#### D. FOR MINERALS NOT EASILY DEPRIVED OF THEIR WATER.

When minerals are present which do not give up their water wholly, even over the blast, as talc, topaz, chondrodite, staurolite, etc., Penfield's simple combination of fire-brick and charcoal oven, depicted in fig. 13, must be used, either with or without a retainer for fluorine, as circumstances demand. The part of the tube in the fire is to be protected by a cylinder of platinum foil tightly sprung about its end,

d the part outside by asbestos board, as well as by wet cloth or paper. A piece of charcoal is likewise laid on the tube, as well as beneath and behind, and the blast flame is given a horizontal direction, so as to play upon the side of the apparatus. In this way a most intense temperature can be reached.

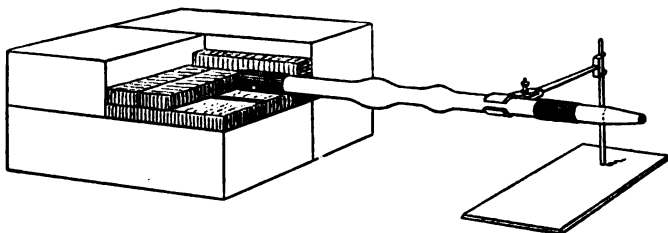


FIG. 13.—Penfield's fire-brick and charcoal oven for use in determining water.

In whichever way the apparatus may be used, the water found is total water, from which that found separately at  $105^{\circ}$  may be deducted if desired.

### C. DIRECT WEIGHING OF THE WATER IN ABSORPTION TUBES.

#### a. PENFIELD'S PROCEDURE.

The simplest of these methods as to apparatus, and one permitting, by the use of auxiliary arrangements, such as are shown and described on page 58, the determination of the hygroscopic as well as any other fraction of the water, is the following glass-tube arrangement (fig. 14) of Penfield,<sup>a</sup> whereby the brick and charcoal oven already referred to (fig. 13) comes again into play, but with the half brick shown in that figure.

The tube is of about 15 mm. internal diameter, and is fitted with two platinum cylinders at A, one inside, the other outside, where the heat exposure is to be most intense.

These are made from pieces of platinum foil, about 0.07 mm. in thickness and 8 by 11 mm. in diameter, which have been previously bent around glass tubes of such a size that when applied to the combustion tubing the spring of the metal will hold them in place.



FIG. 14.—Tube for water determination according to Penfield. A, Outer protecting covering of platinum foil. A second similar foil on the inside prevents the glass from collapsing when heated to redness. b, Cross section of platinum boat.

The platinum boat, 7 to 8 cm. long and 11 to 12 mm. in diameter, with a cross section b, should be used, since this will readily hold a gram of mineral mixed with 5 grams of sodium carbonate. \* \* \* The tube is placed in the angle formed by the charcoal oven, some pieces of charcoal are placed at the sides in front, leaving an opening through which the flame may be directed, and an additional piece is laid on top. The tube can

<sup>a</sup> Am. Jour. Sci., 3d ser., vol. 48, 1894, p. 37, Zeitschr. anorg. Chemie, vol. 6, 1894, p. 22.

readily be brought to a full white heat, and by forcing a slow current of dry air through the apparatus the carbon dioxide resulting from the decomposition can be removed and the water carried over into the weighed absorption tube. The glass fuses between the platinum casings, and in a number of experiments that have been tried there has not been a single instance where the glass tube has broken or shown any indication of breaking. After heating the tube will not crack if it is left to cool slowly on the charcoal, but it can not be used a second time. \* \* \* At the high temperature to which the glass is subjected it of course becomes very soft and the ends must be properly supported; also the rubber connections and absorption apparatus must be carefully screened by asbestos board. By constructing a cover for the boat no material need be lost by spattering, and after making the water determination the contents may be used for the remainder of the analysis.

The inner cylinder of platinum serves to prevent the glass from collapsing as it softens, whereby distortion of the boat would result and its withdrawal for further examination of its contents would be impossible.

#### b. GOOCH'S APPARATUS.

Of more elaborate apparatus, designed to be used with fluxes, the tubulated platinum crucible invented by Doctor Gooch<sup>b</sup> is capable of affording most excellent service, and it is the one by which far the larger number of water determinations in this laboratory have been and are made.

Fig. 15, which hardly needs detailed description, shows it in a modified form, which differs from the original forms of Gooch in that the tubes for connecting with both the drying and absorption vessels are constructed wholly of platinum instead of lead glass. The form of apparatus depicted in fig. 15 differs from that given in Bulletin No. 176 by a considerable shortening in length of the inlet and especially the outlet tube, whereby the cost is reduced and attachment can be made to a glass-stoppered U tube, which is the most satisfactory form for absorption tubes. With tubes of the lengths shown in the figure there is absolutely no danger of their ends becoming hot enough by conduction to scorch or soften the rubber connection.

The extra first cost of the platinum extension to these tubes over the lead-glass ends of Gooch's original and modified forms need hardly enter as a factor into the question of employment of this apparatus. The glass ends often break, and only a rich lead glass, not easily obtainable, can be used, since it alone will not crack at the joint with the platinum after cooling.

As an adjunct to its convenient use there is needed an ordinary upright iron ring stand, with two small sliding rings, and a sliding ring burner provided with entering ducts for gas and air blast. Across the uppermost ring there is an arrangement of stout platinum wire (S, fig. 16), forming at the center of the ring a secure seat

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<sup>a</sup> *Am. Chem. Jour.*, vol. 2, 1880, p. 247; *Chem. News*, vol. 42, 1880, p. 326.

for the upturned flange of the crucible proper. Both rings and burner can be clamped firmly at any height.

The rock powder, having been placed in the cylindrical crucible (C, fig. 15), is there mixed with not more than 3 or 4 grams of fully dehydrated sodium carbonate,<sup>a</sup> or more of lead chromate if carbon is to be likewise determined. The crucible is sunk in its seat, S (fig. 16), in the upper ring, R', and the tubulated cap, T (fig. 15), is fitted on and attached to the calcium-chloride drying towers—preceded by

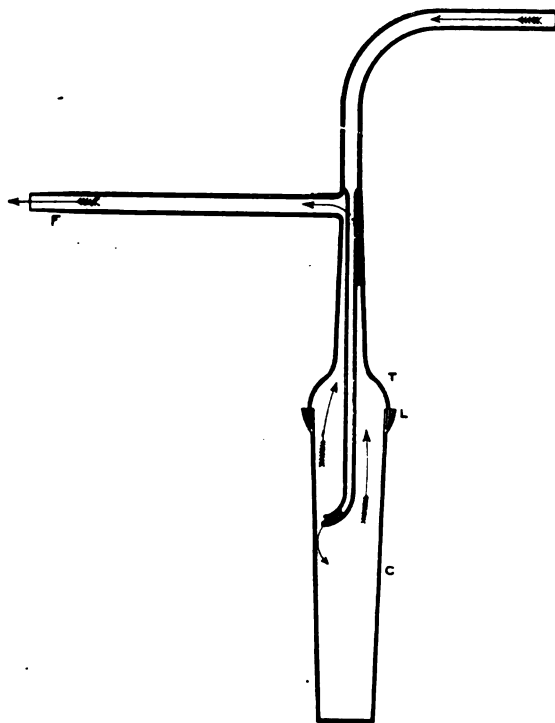


FIG. 15.—Modified form of Gooch tubulated platinum crucible for the determination of water, one-half natural size. Weight about 75 grams.

one containing potassium hydroxide if carbon dioxide is likewise to be estimated—on the one side, and to a sulphuric-acid bulb tube, B (fig. 17), on the other. Powdered sodium tungstate—free from arsenic, which would soon ruin the crucible lips—is now poured into the flanged lip, L (fig. 15), in which the cap rests, and a metal vessel of cold water having been raised up by the lower ring, R'' (figs. 16 and 17), until the platinum crucible is sufficiently immersed, the flame of

<sup>a</sup> This has been heated for a length of time to near its fusing point over a free flame or in an air bath, to decompose the bicarbonate it usually contains, and then placed in a desiccator. Thus heated it is not very hygroscopic. Penfield found that 2.5 grams of it, spread out on a watch glass, gained only 0.0002 gram in fifteen minutes. Potassium carbonate and potassium-sodium carbonate are too hygroscopic by far to be available.

an ordinary blast lamp is turned on to melt the tungstate. As soon as this is fused the flame is removed and the salt solidifies and makes an air-tight joint, the test of which is the permanence of the column of sulphuric acid in the bulb tubes caused by the contraction of the air in the platinum apparatus as it cools.

After drying by a current of air at  $105^{\circ}$  for two hours, more or less (see below, p. 66), by means of an air or toluene bath, as shown in fig. 16, the absorption tube, A (fig. 17), is interposed between the sulphuric-acid bulbs and the apparatus, and, while a slow current of

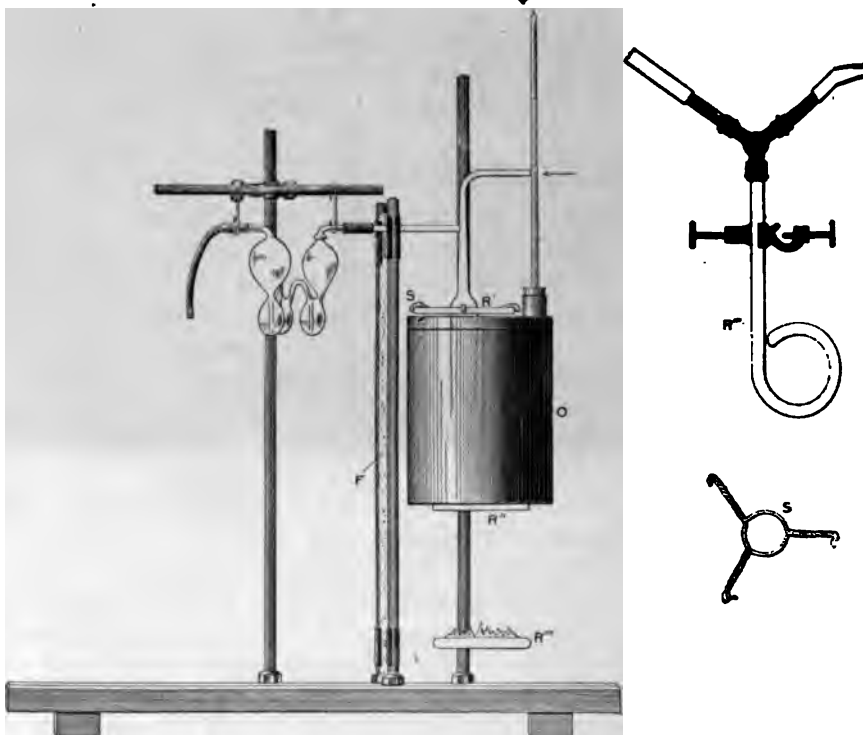


FIG. 16.—Arrangement, during drying, of Gooch apparatus for determining water. S, Seat of stout-platinum wire resting on ring R' and serving as a support for the crucible; R''', blast-fed ring burner; R'', support for air or toluene bath, O; F, asbestos-board shield.

air continues to pass, the gradual heating and subsequent fusion of the flux is brought about by the blast-fed sliding ring burner, R''' (figs. 16 and 17). The sodium-tungstate joint is shielded from the flame by small pieces of asbestos board, P (fig. 17), cut out so as to fit the crucible. When fusion is complete, as shown in the case of sodium-carbonate flux by the decided slackening of the gas current through the safety bulbs attached to the drying tube, the flame is extinguished and a current of air is allowed to continue until the apparatus is cold.

This apparatus suffers from the drawback of being slightly permeable to combustion gases at high temperature. The defect can be overcome by causing the flame to play upon an outer ordinary platinum crucible, kept permanently filled with sodium-potassium carbonate. This protective crucible, however, is soon ruined for other purposes, being distorted by the alternate expansion and contraction of the carbonate.

It has been found that if the operation is carried out expeditiously and the final full heat applied for but a few minutes the error due to

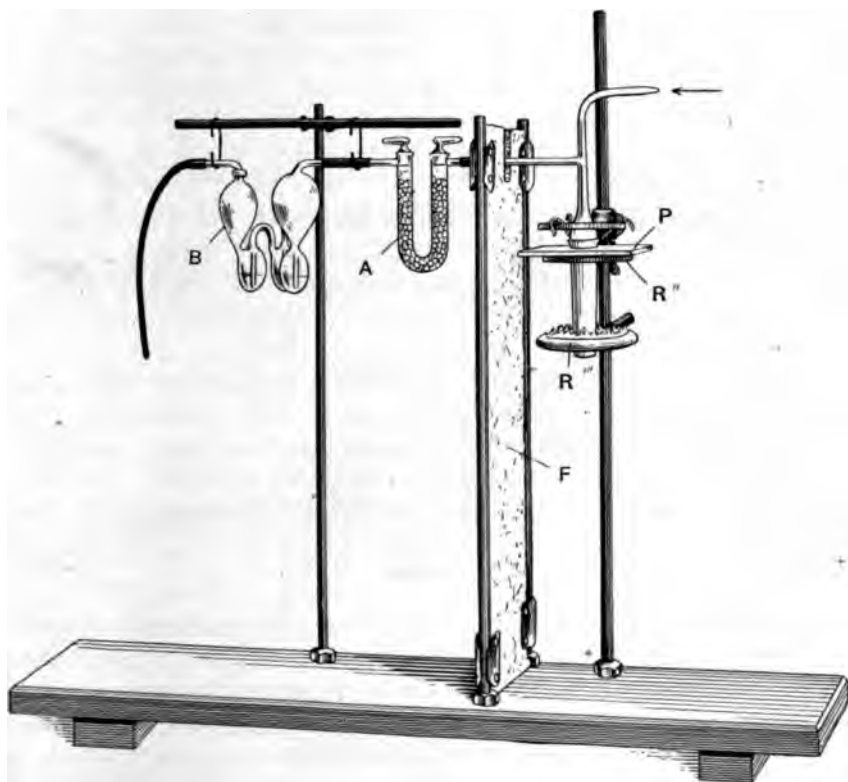


FIG. 17.—Arrangement, during fusion, of Gooch apparatus for determining water. R''', Blast-fed ring burner; P, protective asbestos shield resting on ring R''; F, asbestos-board shield; A, calcium-chloride tube; B, sulphuric-acid bulbs serving to show the rate of gas flow through the absorption tube and at the same time to prevent back entry of moisture from the air into A.

penetrating water gases is inappreciable. This hastening may be rendered safer by using rather finely powdered calcium chloride in the central section of the U-shaped absorption tube to avoid large air channels. Through this or any other apparatus based on similar principles the air current should always be forced, not drawn. A warm air blast directed upon the exit tube near its entrance into the absorption tube greatly shortens the time required and is to be recommended.

In this apparatus only the water expelled above  $100^{\circ}$  to  $110^{\circ}$  should as a rule be determined, and to effect drying of the mixed mineral powder and sodium carbonate, after luting the tubulated cap on the cylindrical crucible with sodium tungstate, the tube is sunk through a round hole in the cover into a small cylindrical air bath (fig. 16), which can be heated from beneath by the same ring burner which is subsequently to fuse the flux. A slow current of air is then forced through and the drying satisfactorily accomplished.

The reason why it is unsafe to attempt estimation of "hygroscopic" moisture in this apparatus is that the luting of the two parts must be done by direct application of a flame to the tungstate, and considerable water vapor may enter the apparatus and be in part retained by the dried sodium carbonate.

#### C. MERITS OF THE ABOVE FORMS OF APPARATUS.

The apparatus described in *a* and *b* permit the determination of other constituents besides water in the same portion if necessary, and by the use of lead chromate instead of sodium carbonate, graphite, or the carbon of organic matter, can be simultaneously determined with the water.

To one accustomed to its use, and with a drying and suspension attachment permanently set up, the Gooch apparatus, considering its limitations above set forth, offers perhaps the most handy and convenient means for the determination of water in rocks. Its high first cost, in comparison with the glass tube, is fully made up in time by its durability.

#### d. JANNASCH'S METHODS.

This zealous deviser of methods for mineral analysis has published in the *Zeitschrift für anorganische Chemie* and the *Berichte der Deutschen chemischen Gesellschaft* several papers dealing with the problem of water determination in minerals, and in his text-book<sup>a</sup> these are collected in more or less modified form.



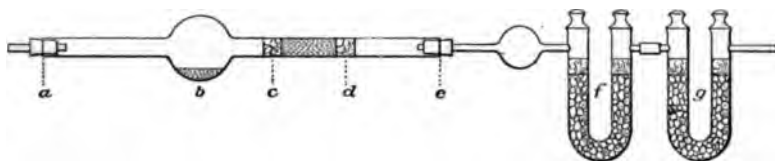
FIG. 18.—Glass tube for determination of water (Jannasch). *b*, Mixture of mineral powder with borax; *c*, *c*, plugs of glass wool; *a*, layer of lead chromate or lead oxide. Total length of the tube, 33 cm; inside diameter, 12-14 mm.

For the majority of silicates he finds dehydrated borax powder a most efficacious flux, usually at a very moderate temperature. The fusion is accomplished either in a platinum boat within a glass tube or in a tube of the form and dimensions shown in fig. 18.

<sup>a</sup> *Praktischer Leitfaden der Gewichtsanalyse*, Leipzig, von Veit & Co. (1897).

rocks or minerals containing not much fluorine a retaining layer of anular lead chromate, or of previously fused and powdered lead, is used as shown at *a*. Plugs of glass wool are used, at *c*, either or not the boat is employed, the borax is first introduced together with the retainer, is thoroughly dried out in an asbestos by a hot-air current. Then, after cooling, the mineral powder is led and thoroughly mixed with the borax. Heat is applied by a flame to the mixture, which soon melts and forms a clear fusion, the action is complete. The blast may be used in extreme cases. The layer of retainer must be kept warm by an auxiliary flame, and the absorption tube must be removed before the flame under the fused mass is extinguished, for the glass breaks as soon as this is done. Carbon dioxide can simultaneously be determined by attaching a soda-lime tube to the calcium-chloride tube. For one-half to 1 gram of substance Jannasch uses  $1\frac{1}{2}$  to 2 grams of dehydrated borax.

Regarding the borax method, its inventor insists on the following as essential to success, especially when the blast can not be used: Most thorough mixing of flux and mineral powder and a most capable fineness of the latter.



—Glass tube for determination of water in special cases (Jannasch). Length from *a* to *e*, 26 cm.; inside diameter somewhat over 1 cm.; volume of bulb *b*, 25 cm.<sup>3</sup>; *c d*, retaining layer of lead oxide in plugs of glass wool. *f*, calcium-chloride absorption tube; *g*, protective tube.

The borax itself is prepared by heating pure crystallized borax in a platinum dish till a small portion has melted. That remaining unfused is powdered and again heated in the dish to dull redness for fifteen minutes, with constant stirring. The powder is placed in a tube with a fitting glass stopper and kept over sulphuric acid. It must be kept long without reheating, because of being hygroscopic. Another form of tube used by Professor Jannasch for special purposes and recommended by Doctor Dittrich is shown in fig. 19. Minerals such as topaz, which is not fully decomposed by the borax alone and which contains a large amount of fluorine, are fused at *b* about six times their weight of lead oxide. A layer of lead oxide (1 part of oxide and peroxide.—Dittrich) between *c* and *d* serves to retain any fluorine escaping from the fusion. Doctor Dittrich uses this form generally for substances which can not be heated in the best form of Penfield tubes and for those which give off their gas on simple ignition, without admixture of lead oxide, though, of course, with the retainer for sulphur, fluorine, etc.



If  $\text{CO}_2$  is to be simultaneously determined, any carbonate in the lead oxide must be first destroyed by heat. As in the preceding description the layer of retainer must be kept continually hot by an auxiliary flame.

## 6. SILICA, SEPARATION FROM ALUMINA, ETC.

### A. ALTERNATIVE METHODS OF DECOMPOSING THE ROCK.

#### a. GENERAL CONSIDERATIONS.

The practice of separating alumina, etc., by the usual methods, after first attacking the rock powder by hydrofluoric and sulphuric acids—silica being estimated in a separate portion—while attractive in principle, was abandoned by me after fair trial, owing to the disturbance sometimes occasioned by incomplete expulsion of fluorine and to a less degree by the presence of sulphates instead of chlorides. With the exception of a comparatively few analyses made thus, the sodium-carbonate method has always been employed. In the case of rocks rich in fluorine strict accuracy would require the separation of silica to be made as in the Berzelius method for fluorine estimation (see p. 155, also footnote, p. 71), but in practice it is not often necessary to resort to this tedious procedure, since the amount of fluorine is usually small and it can by no possibility cause a loss of much more than three-fourths its own weight of silica by volatilization as silicon fluoride when the sodium-carbonate fusion is evaporated directly with hydrochloric acid. Probably the loss is less, since some fluorine perhaps escapes as hydrofluoric acid. However this may be, the error is of comparatively slight importance, since it always attaches to the constituent present in greatest amount.

Various fluxes other than alkali carbonates have been recommended for breaking up silicates insoluble in ordinary acids, such as lead and bismuth oxides, lead carbonate, borax, and boric oxide. Professor Jannasch and his pupils have been especially active in this line of work, as is evidenced by their numerous published papers. One of the advantages most of these fluxes possess over the alkali carbonates is their removability after serving their purpose, thus allowing the various separations to be made more perfectly and without the annoying interference of several grams of foreign fixed salts, which are most troublesome in that part of the analysis devoted to the separation of silica, alumina, iron, lime, and magnesia.

Another of their advantages is that with some of them it is possible to estimate in one portion the alkalies in addition to those constituents usually determined in the silica portion. Where the material is limited, as it so often is in mineral analysis, this is a most important

advantage, sufficient to outweigh all possible objections; but in rock analysis, where the supply of material is usually ample, it is rarely worth considering. A still further point in their favor is that it is probably more easy to obtain them entirely free from fixed impurities than an alkali carbonate.

There are, however, objections to their use. With some of them an extraordinary amount of time must be devoted to grinding the mineral to an impalpable powder, and the flux itself may need considerable hand pulverization. Once introduced, they must be removed before the analysis can be proceeded with, and this removal takes much time and is always a possible source of error.

In mineral analysis these objections are entitled to far less weight than in rock analysis, since the object sought—usually the deduction of a formula—warrants the expenditure of much time and painstaking care. Finally, it has been found that one or more of these fluxes are not available for altogether general use, since certain minerals do not easily succumb to their attack under simple conditions, as andalusite with boric oxide and others with lead oxide (Jannasch). Therefore, however well adapted one or the other of these methods may be for the analysis of homogeneous minerals, it is very improbable that the anticipations of Professor Jannasch, to the effect that the boric-oxide method will soon supersede the alkali-carbonate fusion method in rock as well as in mineral analysis, will be speedily realized. Nevertheless, the boric-oxide fusion method, owing to its evident merit, will be described in detail after brief reference to a means of bringing refractory silicates into solution without employing any solid reagent.

The methods of decomposition and the subsequent treatment of the silica hereinafter described are with few exceptions applicable to definite silicate mineral species as well as to complex mixtures of them.

#### DECOMPOSITION OF REFRACTORY SILICATES BY HYDROCHLORIC ACID UNDER PRESSURE.

Jannasch<sup>a</sup> pours upon the finely ground rock powder contained in a platinum tube of about 26 cm.<sup>3</sup> capacity a somewhat diluted hydrochloric acid (4 acid to 1 water), places over the open end a cap which does not hermetically close the tube, inserts the latter in a larger tube of potash glass likewise partially filled with the diluted acid, seals the glass tube, places it in turn in an inclined position in a steel Lannesmann tube containing ether or benzine to equalize the pressure, and heats to any desired temperature up to 400°.

The chief drawback seems to be a somewhat incomplete decomposition, doubtless due to the necessarily inclined position of the tube, which causes the powder to collect at the lower end, and thus renders the composition less complete than if the material were spread evenly

<sup>a</sup> Ber. Deutsch. chem. Gesell., vol. 24, 1891, p. 273; Zeitschr. anorg. Chemie, vol. 6, 1894, p. 72.

throughout the length of the tube. Further, the acid strongly attacks the platinum unless the air in both the platinum and the glass tubes is replaced by carbon dioxide. Even when this is done, several milligrams of platinum are found in the silicate solution.

Nevertheless, to those possessing the necessary platinum and steel tubes the method can render efficient service in special cases when economy of material is imperative.

#### C. THE BORIC-OXIDE METHOD OF JANNASCH AND HEIDENREICH.<sup>a</sup>

*a. Preparation of the boric oxide.*—This demands, if the alkalis are to be estimated in the same portion as silica, etc., an absolutely alkali-free boric acid, which can be prepared by two or three recrystallizations of a good commercial article. The purified crystals are dehydrated and fused in a large platinum crucible. This is then suddenly cooled to cause the anhydride to crack into pieces of a size convenient for powdering, which are to be kept in a tight glass and powdered as needed, since the anhydrous oxide is hygroscopic.

*β. Treatment of easily decomposable silicates.*—To this flux Jannasch and Heidenreich find that nearly all silicates readily succumb over the ordinary blast lamp. The fusion is made in a large crucible holding 40–65 cm.<sup>3</sup>, and the proportion of flux to be used is gaged according to the nature of the silicate, ranging from 3 to 8 and more parts to 1 of mineral. This last must be finely powdered, especially the most resistant; the authors recommending the expenditure of one-half to one hour's time for the grinding of one-half to 1 gram of powder. A low burner heat is applied for five to ten minutes till water is expelled; the heat is then gradually increased till the gas is fully turned on. Bubbling and rising in the crucible is prevented as far as possible by using a short platinum rod which does not reach above the edge of the crucible. When the mass has been in quiet fusion for a time in the covered crucible the blast flame is applied. The average duration of the entire operation is twenty to thirty minutes, but depends much on the character of the mineral.

*γ. Treatment of refractory silicates.*—For those minerals which, like andalusite, cyanite, and topaz, are not fully decomposable by the heat of the ordinary blast flame, Jannasch and Weber<sup>b</sup> use a flame fed by oxygen instead of air. The blast lamp, of 2½ mm. opening, is supplied with gas from at least five or six ordinary gas cocks, and the flame is made broad and free from luminosity. The mineral having been first heated as above described, but with a much larger proportion of flux—as high as 30 to 1—a few grams additional of boric oxide

<sup>a</sup> *Zeitschr. anorg. Chemie*, vol. 12, 1896, p. 208. This method of decomposing rocks with a view to determination of their contained alkali originated with Sir Humphrey Davy, as shown in his paper "On a method of analyzing stones containing fixed alkali by means of the boracic acid" (*Phil. Trans.* 1805, p. 231; *Ann. d. chim.*, vol. 60, 1806, p. 294; *Gilbert's Annal.*, vol. 30, 1808, p. 369; *Tulloch's Phil. Mag.*, 1806, p. 146). Original with Jannasch and Heidenreich is the manner of getting rid of the induced boric oxide.

<sup>b</sup> *Ber. Deutsch. chem. Gesell.*, vol. 32, 1899, p. 1670.

are added and the oxygen blast is applied till, in ten or fifteen minutes, the fusion is as transparent as glass.<sup>a</sup>

δ. *Further treatment after fusion.*—From this point the further treatment is the same in both cases and, as modified by Jannasch and Weber<sup>b</sup>, is as follows:

The hot crucible is cooled in cold water and the contents are turned into a very large porcelain or platinum dish, to which, after covering with a glass, a saturated solution of hydrochloric-acid gas in methyl alcohol is added.<sup>c</sup> The cover being then removed, the liquid is heated to boiling, over asbestos board, by an inch-high flame, with constant stirring, or it is left without attention over a lower flame or on a water bath heated short of boiling. The crucible is cleansed in a similar manner, and its contents are added to the dish. In ten to fifteen minutes, with occasional addition of the methyl chloride, solution is complete and the liquid is then boiled down to a small volume and evaporated to dryness on the bath. The residue is then digested on a bath at 80° to 85° three or four times in succession with the ether solution, in order to remove the last traces of boron as boric ether. Care should be taken to wash down from the sides of the dish, with methyl-chloride solution, the boric acid formed and deposited thereon during the evaporation.

ε. *Possible objections to the boric-oxide method.*—Very much is claimed by Jannasch for this method, but with all its undoubted merit there are two points which may militate against it. The boric ether, driven off in such quantities, at once decomposes in contact with moisture, and boric acid settles over all objects with which it comes in contact. The hood must become thickly coated. Hence a special hood for these evaporations alone seems to be called for, otherwise boric acid may at any time fall into other dishes and cause untold trouble. The second objection attaches to the use of the oxygen flame when alkalies are to be estimated in the fusion, and the ability to so determine them is one of Jannasch's chief claims in favor of the method, for it can not be doubted that at the high temperature of his flame alkalies are volatilized. Borax can be slowly but wholly volatilized over the ordinary blast, hence there is great reason to fear sufficient loss at this much higher temperature to give rise to serious error at times.

#### D. THE SODIUM-CARBONATE METHOD.

α. *Advantages of sodium carbonate over sodium-potassium carbonate.*—Except in special cases, as when fluorine or chlorine are to be

<sup>a</sup> An interesting and important observation reported by Jannasch and Weber is that when the oxygen blast has been used for silicates carrying fluorine or mixed with fluorides the fluorine seems to be wholly expelled as boric fluoride without loss of silica. If this should prove to be generally true, an easy way is at last afforded for determining silica in such cases, where even its detection, when present in small amount, has heretofore been difficult.

<sup>b</sup> Ber. Deutsch. chem. Gesell., vol. 32, 1899, p. 1670.

<sup>c</sup> Made by passing dry hydrochloric-acid gas into cooled methyl alcohol for from one to two hours.

determined, there is no advantage in using the much recommended, because more fusible, double carbonate of sodium and potassium, or the equimolecular mixture of the normal carbonates, or of sodium carbonate and potassium bicarbonate. As Dittrich says,<sup>a</sup> potassium salts are more prone to pass into precipitates than sodium salts and it may be that the higher melting point of sodium carbonate is a distinct advantage. Certainly, for effective decomposition of some rock constituents, a far higher temperature than that of the fusing point of the double salt is required.

*β. Purity of the sodium carbonate.*—Notwithstanding the most earnest efforts for years, it has been impossible to procure, either in the open market or by special arrangement with manufacturers, a grade of sodium carbonate which can be called chemically pure. With special precautions small lots can be prepared in the laboratory that will contain less than 1 milligram total impurity in 10 grams; but such an article can not be purchased in the market, and rarely will the so-called chemically pure dry sodium carbonate contain as little as 1 milligram in 10 grams. The invariable contaminating substances—aside from sand and straw, which have sometimes been found in large amount—are silica, alumina, iron, lime, magnesia, and sometimes phosphoric oxide, all of these going into aqueous solution with the carbonate. The chief of these impurities are usually silica, alumina, and lime. An article of the above degree of purity is satisfactory in almost all imaginable cases, since the use of the usually extravagant amount of 10 grams for a fusion would introduce an error of but 0.1 per cent in the analysis, supposing 1 gram of mineral to be operated on, and it would, moreover, be distributed over several constituents. This error is undoubtedly fully equaled by the introduction of dust from the air in the various long evaporations.

It is to be borne in mind that the so-called dry sodium carbonate, "C. P.," almost always contains some bicarbonate and hence yields water on heating. This, however, in no way detracts from its usefulness, and it is not necessary to convert it wholly to the normal salt for fusions of the kind now contemplated. In fact, C. Holthof<sup>b</sup> most strongly recommends using the bicarbonate itself as a flux instead of the normal carbonate (see *δ*, p. 74).

*γ. The fusion with normal sodium carbonate.*—Ordinarily from 4 to 6 parts of the flux should be used to 1 of rock powder, thoroughly mixed in a crucible of 20 to 30 grams weight. It is inadvisable to use the much larger proportion of flux recommended by some writers, except as it may be shown in isolated instances to be needed. The larger amounts introduce more impurity than the smaller and necessitate longer washing of precipitates. At first the crucible (covered) is

<sup>a</sup> *Anleitung zur Gesteinsanalyse*, 1905, p. 5.

<sup>b</sup> *Zeitschr. anal. Chemie*, vol. 23, 1884, p. 499.

placed over a moderately low Bunsen flame, which is gradually increased to the maximum and maintained there till the mass is incipient. There should be no violent action. The contents of the crucible will then appear, in the case of highly feldspathic or quartzose rocks, as a viscous liquid, occasionally almost clear, though generally more or less turbid, and when placed over the blast little or no further effervescence occurs. Melts of this character disintegrate very readily in water. With less siliceous rocks the fusion is less perfect and may be far from complete, though this does not necessarily imply incomplete decomposition of the silicates. It is generally advisable and often necessary to place these less fusible mixtures over the blast lamp, when a further and very marked escape of carbon dioxide takes place, and the crucible cover should be cautiously lifted at intervals to avoid loss by boiling over. This boiling is due not merely to further action of the carbonate on the rock-forming minerals, but oftentimes more to the decomposition of the alkaline-earth carbonates, whose oxides then enter into combination with other constituents of the flux and rock to form compound silicates and probably aluminates. It is a great mistake to regard, as many seem to do, the alkaline-earth oxides—magnesium, iron, and manganese—as present in the form of carbonates after a blast fusion. They are rarely in that state even when only the Bunsen flame has been used.

The blast flame during fusion should not be directed vertically against the bottom of the crucible, but at an angle against the side and bottom, nor should the flame be allowed to envelop the whole crucible. These precautions apply in all ignitions of reducible substances, and yet they are rarely observed. In neither case, if neglected, will there be a necessary oxidizing atmosphere within the crucible; on the contrary, reduction may occur fraught with serious consequences. This is especially true if the rock contains more than traces of pyrite or iron sulphide, when, after cleansing and igniting the crucible, there may appear on its interior a darkening due to oxidation of reduced iron which had alloyed with the platinum. This may in exceptional cases amount to several milligrams in weight, and can be removed easily by repeated ignitions, followed each time by scouring or treatment with hydrochloric acid or acid potassium sulphate. In order to avoid the use of niter in case of pyritiferous rocks, it is well to first dust gently the weighed powder in the crucible in which the fusion is to be made, turning the crucible around a few times to expose all of the powder to the air. With rocks exceptionally high in pyrite the roasting is best done in a porcelain crucible. In such case, after transferring the greater part to the platinum crucible, if brushing with a camel's-hair brush does not remove the last of any adhering dust, pouring with a little sodium carbonate will be effective.

It sometimes happens that the cooled flux, and even its solution,

will indicate absence of manganese when it is really present in quantity to give normally a strong coloration. Two fusions made side by side or successively, under apparently similar conditions, may in one case show little or no manganese, in the other considerable. This observation has been frequently made, and therefore the absence of a bluish-green color in the fusion is not to be taken as proof of the absence of manganese. This difference of behavior I can ascribe to no other cause than that of a reducing atmosphere in one of the crucibles and an oxidizing one in the other, even though the conditions were apparently alike. It is, of course, not to be expected that the green color can show until all other oxidizable components of the rock, like sulphides, ferrous iron, and organic matter have been fully oxidized, which, however, is soon the case if air has access to the surface of the melt.

δ. *Fusion with sodium bicarbonate.*—C. Holthof, who recommends<sup>a</sup> this flux strongly, uses 12 to 15 parts of it to 1 of the sample. One quarter of this is placed on the bottom of the crucible, a second quarter is thoroughly mixed with the rock powder in a small warmed dish, and the mixture is then mixed with a third quarter on glazed paper and poured into the crucible. The last quarter is used for rinsing the dish and paper and covering the contents of the crucible, which may be half full. The crucible is heated over a low flame till the bottom is dull red, then during fifteen minutes the flame is increased till the lower quarter of the crucible is dull red, in which state it is maintained for fifteen minutes. Then fusion is brought about by the full flame and maintained for a time. He claims that action between the silicate and carbonate takes place at a lower temperature than with the normal carbonate before fusion, and that for this reason there is almost no spattering onto the lid of the crucible.

ε. *Treatment after fusion.*—When fusion is complete, the crucible is seized with the tongs (fig. 1, p. 28), and the contents are caused to solidify in a thin sheet over the sides and bottom by imparting an appropriate gyratory motion with the arm during the cooling process. This is preferable to allowing the melt to form a thick cake at the bottom, since much less time is required for disintegration, and separation from the crucible is usually much easier.

The contents of the crucible are placed in a rather tall covered beaker with some water, and hydrochloric acid of 1.1 specific gravity is gradually added in excess. The depth of the evanescent pink color usually produced on addition of the acid allows of judging approximately the amount of manganese present. The beaker is placed on the water bath, and when disintegration is complete, having been assisted by gentle pressure with a blunt glass rod, the contents are transferred to a large platinum dish and evaporated on the bath. It is permissible to dispense with the beaker and to perform the whole

<sup>a</sup> Zeitschr. anal. Chemie, vol. 23, 1884, p. 493.

operation of solution in the platinum dish, but there will be greater solution of platinum in this case by reason of the action of the hydrochloric acid on sodium manganate, etc., in direct contact with platinum.

#### B. SUBSEQUENT TREATMENT.

From this point the treatment will ordinarily be the same whether the boric-oxide or the sodium-carbonate method of decomposition has been employed.

##### a. SEPARATION OF SILICA.

*α. Reasons for adoption of accepted procedure.*—The once universal practice of employing a single evaporation with hydrochloric acid for the dehydration of silica has been thoroughly discredited by the work of a number of writers. R. Bunsen<sup>a</sup> was aware of the impossibility of separating silica by a single evaporation, and he corrected the alumina in his silicate analyses for the silica it was always found to contain. Later, E. Ludwig,<sup>b</sup> and still later, C. Meineke,<sup>c</sup> drew renewed attention to the fact and insisted on the importance of correcting the alumina in the manner indicated. Ludwig and, at first, Meineke contented themselves with a single evaporation, and maintained that all of the unprecipitated silica was to be found with the subsequently precipitated iron and alumina. In his second paper Meineke prescribes two evaporations (presumably with intervening filtration) and the rejection of the silica subsequently recovered from the alumina as due to impurity in the ammonia or derived from the vessels, unless these were of platinum. He furnishes quantitative data.

In spite of these publications the practice continued of employing but a single evaporation. Some years later Alexander Cameron,<sup>d</sup> seemingly in ignorance of the earlier work, reopened the subject with additional quantitative data, and insisted on more than one evaporation with intervening filtration instead of trying to recover the balance of the silica from the alumina. He also showed, as Ludwig had asserted, that a common practice of evaporating to dryness several times with fresh portions of acid without intervening filtrations did not reduce the silica in the filtrate, and that the presence of aluminum, iron, and calcium was without influence on the results; further, that the dehydration was more complete when a high temperature was employed, but that it could not be made complete at one treatment by any modification of the process.

According to J. P. Gilbert,<sup>e</sup> drying temperatures above that of the steam bath offer no advantage unless much magnesium is present, when the most favorable temperature is 120°. He found that much

<sup>a</sup> Ann. Chem. Pharm., vol. 61, 1847, p. 265.

<sup>b</sup> Zeitschr. anal. Chemie, vol. 9, 1870, p. 321.

<sup>c</sup> Repert. anal. Chemie, vol. 7, 1887, pp. 215, 757.

<sup>d</sup> Chem. News, vol. 60, 1894, p. 171.

<sup>e</sup> Technology Quarterly, vol. 3, 1890, p. 61; Abstract in Zeitschr. anal. Chemie, vol. 29, 1890, p. 688.



calcium chloride seems to facilitate dehydration of the silica, but that magnesium chloride above  $120^{\circ}$  by decomposing forms a silicate which dissolves in hydrochloric acid and increases the amount of silica carried into the filtrate. He confirmed the earlier belief that drying temperatures higher than that of the steam bath increase the amount of insoluble impurity in the silica, and that this amount can not be overcome by long digestion with hydrochloric acid. Further, he confirmed D. Lindo's statement<sup>a</sup> that evaporation with sulphuric acid till the appearance of white fumes gives a higher result in silica than with hydrochloric acid. But for general rock analysis the use of sulphuric acid at this stage must be rejected utterly.

Some of the above experiments of Cameron and Gilbert were repeated and confirmed by me.<sup>b</sup> In the course of this work it was found that hydrochloric acid itself exerts a marked solvent action on silica that has been separated from solution in any of the ways above outlined, whence it becomes plain how a portion of the silica always found in the filtrates gets there, and that it is hopeless to expect to prevent this by a single prolonged drying. Other explanations commonly offered for the observed solubility are the formation of soluble silicates by interaction between the silica and salts present during drying and the protective influence of those salts. The former of these should become more active with increasing temperature, though Gilbert's work does not seem to indicate this except when magnesium is present in quantity.

C. Friedheim and A. Pinagel<sup>c</sup> prescribe washing the silica with dilute hydrochloric acid instead of hot water because of the marked solubility in water observed by them. But, according to experiments made by myself to test this point, the direction is a vain one, for the results obtained by following it in the ordinary course of rock analysis were worse, if anything, than when pure water was used.

*β. Procedure in absence of notable amounts of fluorine.*—Based on the above observations the following procedure is deemed best adapted for general rock analysis, in the absence of more than 0.2 or 0.3 per cent of fluorine.

The evaporating dish should be of platinum and as large as possible to permit considerable surface distribution of the dry mass. Porcelain may be used, but never glass, if platinum is not available. Water or steam-bath temperature suffices, is indeed demanded, for the evaporation to approximate dryness. If the analysis is begun in the morning the first filtration can be made in the late afternoon. Nothing is gained by continuing the evaporation long beyond apparent dryness this first time, for the slight reduction in the soluble silica

<sup>a</sup> *ibid.* 60, 1889, p. 11.

<sup>b</sup> Fraction of silica; *Jour. Am. Chem. Soc.*, vol. 24, 1902, p. 262; *Chem. Abstracts* referred to here and on several of the follow-

ned by so doing is offset by a large loss of time, nor is there much vantage in crushing the residue to powder, whereby, moreover, the h is easily scratched. The amount of silica passing into the filtrate l generally vary from 1 to 3 per cent of the amount present, and out little less after twenty hours drying than after one-tenth of that ~~as~~<sup>a</sup>. Upon the seemingly dry mass is poured enough strong hrochloric acid to thoroughly drench it, then an equal bulk of water, l the dish is placed covered on the bath for ten to thirty minutes l the contents occasionally stirred. It is quite permissible and some- es advisable to defer adding the water till the strong acid has been contact with the salts for ten or fifteen minutes. More water is then led and filtration proceeded with, first by decantation, leaving nearly the silica in the bottom of the dish. It is generally in a coarse idition and may be ground finer with a pestle. If there is a good l of iron in the rock it may be advisable to add hydrochloric acid half strength and heat, after which the silica is brought onto the er. It is not necessary to remove at this time the film of silica t may adhere to the dish in spots beyond the power of a stiff ther to remove, for in the subsequent evaporation it will disap- ar. The washing should be done with cold water or with hot ute acid till the absence of any yellow color in the precipitate or per indicates removal of most of the iron. Hot water may with vantage be used for the last washings, and the paper should be ked dry at the pump.

The filtrate is evaporated again in the same dish to dryness. Usuy this state will be reached by the following morning. It may be stened, if desired, by placing the dish in an air bath at 110° or 120°. is probable that a much higher temperature would in the majority cases have no bad effect except to increase the amount of insoluble tter other than silica. If the data of experiments 6 to 9 in the per already cited<sup>a</sup> are conclusive, they show that at this stage ough drying exercises an appreciable effect in reducing the silica in e next filtrate, though the small amounts there reported when experi- nting with pure quartz are not to be expected in rock work. If ne is no object, and great accuracy is sought, a third evaporation and ration should be made, for the silica in the second filtrate will ount to 2 to 4 milligrams. Nearly the whole of this can be recov- d later from the alumina, but not all, hence the advisability of uring as complete separation here as possible. The second and if ed be third filtrations with their attendant operations consume s time than the first, and, of course, smaller filter papers are to be ed. It is to be noted that these later silicas are more colored than e first, and if the rock is even moderately titaniferous are by no ans pure silica.

<sup>a</sup> See Jour. Am. Chem. Soc., vol. 24, 1902, p 366.

With minerals like the zeolites, or artificial products such as Portland cements, which are directly soluble in hydrochloric acid, the drying may be very much hastened by placing the covered dish, after bringing to approximate dryness on the bath, on a sand bath or on a triangle which itself rests on a hot plate. The temperature may rise in the dish to 200°; in fact, according to Bertram Blount,<sup>a</sup> it should not be lower. After an hour's exposure to this temperature less silica will perhaps pass into the filtrate than after prolonged drying at steam-bath temperature, but this is usually at the expense of greater contamination of the filtered silica. Since this contamination is very large (see e, p. 80) with highly titaniferous and ferruginous rocks and minerals, the operator should use discretion in his choice of a high drying temperature.

*γ. Procedure with rocks and minerals containing fluorine.*—This subject has been touched on in commenting on the boric-oxide and sodium-carbonate methods of fusion,<sup>b</sup> and will be considered in detail under the head of "Fluorine" (p. 155).

#### b. IGNITION OF SILICA.

The application of a strong blast<sup>c</sup> for twenty to thirty minutes with crucible covered is absolutely necessary to expel all moisture from the silica, and the latter is then not hygroscopic. The first of these statements, long extant in the literature, has been disputed by G. Lunge and C. Millberg<sup>d</sup> on the basis of experiments made on silica obtained by the decomposition of silicon tetrafluoride by water. It has been shown,<sup>e</sup> however, that, while the objection holds good for silica thus prepared, it does not for silica obtained by the decomposition of an alkali silicate by means of an acid. F. P. Treadwell has confirmed the observation as to the difference in behavior of the two forms of silica, but regards the blast as unnecessary if a Teclu instead of a Bunsen burner is used. My experience is not in agreement with this.

Although the above time limit usually suffices with a powerful blast, and may be materially shortened for small amounts of silica, such as

<sup>a</sup> Jour. Soc. Chem. Ind., vol. 21, 1902, p. 1217.

<sup>b</sup> See a, p. 68, and footnote, p. 71.

<sup>c</sup> It must be borne in mind that some platinum crucibles lose weight steadily and very appreciably on long blasting, not only when new but even after long use. When a crucible suffers from this defect the rate of loss should be ascertained from time to time and allowance made accordingly, or else the weight of the crucible should be taken after and not before ignition of the precipitate. (See on this subject Hall, R. W., Jour. Am. Chem. Soc., vol. 22, 1900, p. 494.) It was hoped that with the advent of the electric furnace prolonged ignitions would not be subject to this source of error, but it was soon found that at 1,250°, the maximum temperature attainable in the furnace at the Survey, medium-sized crucibles lost approximately 1 milligram in weight per hour. Similar losses have since been noticed and published by others. Their cause seems to be chiefly the distillation of iridium out of the alloy. At one time an error of about the same magnitude in the opposite direction caused perplexity until it was found that the platinum wire in the bed-plate of the furnace was burned out. The crucible, then occupying the coolest place in the furnace, received a deposit of some of the metal volatilizing from the wires in the side plates.

<sup>d</sup> Zeitschr. angew. Chemie, 1897, p. 425.

<sup>e</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 24, 1902, pp. 372-373.

sually found in limestones and even in cements, there is need ionally to prolong it in very exact work until repeated weighings the same value or a loss no greater than that suffered by the crucible itself.<sup>a</sup>

#### C. CORRECTION FOR IMPURITIES IN THE SILICA.

e weight of the crude silica should always be corrected for rities, which are never entirely absent, by evaporating with uric and hydrofluoric acids and again blasting for a minute or according to the amount of residue. It is a mistake to neglect er weighing at this point, even if the residue may appear insignificant. In the author's experience it never has been found to be ighable, and, furthermore, the error due to loss in weight of the ole itself is eliminated if the weighing is made. The silica in the ole should be moistened with water before adding the hydro-c acid, though with strongly blasted silica the action is not so vio-as with that which has not been thus treated. With rocks ing 60 to 80 per cent of silica one or two drops of sulphuric acid d one-half suffice, but with less siliceous rocks the amount ought increased, not only in order to surely afford acid enough for the rsion of all the contaminating bases to sulphates, but also to nt loss of titanium by volatilization as fluoride, since with singly basic character of the rocks the percentage of this ele-in the residue is likely to increase.

ecipitated silica is easily removed by one evaporation with a ency of hydrofluoric acid, but if quartz happens to be present ; not the case, for it is but slowly dissolved and more than one nent may be necessary.

; subsequent precipitate of alumina, etc., is usually ignited in ucible containing the residue from the silica.

#### d. ACCURACY OF THE SILICA DETERMINATION.

observing the above rules—use of platinum, repeated evapora-and filtrations, proper blast ignition, and correction for foreign r—the determination of silica, from being one subject to grave has become one of the most exact of which I have cognizance, ded precautions are taken to recover, as hereinafter described, ortions that inevitably are to be found with the alumina. With il work variations of 0.1 per cent are the exception in duplicate ses.

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be proof see Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 24, 1902, p. 373. The duty of every to test the efficiency of his blast or muffle can not be too strongly emphasized. The discordance / statements and results on a variety of subjects is to be attributed to a difference in the igni- perature employed by different writers. A really good blast will readily melt a few centigrams class powder in the bottom of a 15-gram platinum crucible, showing a temperature of prob- 10°.

## c. COMPOSITION OF THE RESIDUE OBTAINED FROM THE SILICA.

The qualitative composition of this residue varies with different rocks less than its quantity. It will contain alumina and ferric, titanic, and phosphoric oxides invariably if these are present in the rock. If the rock is low in the last three, particularly the last two, the residue should be slight, occasionally less than 1 milligram. It is usually much greater, and may amount to 2 or even 3 per cent with basic rocks very rich in titanium and phosphorus. It is, however, a great mistake to suppose, as some chemists still seem to, that all or nearly all the titanium is to be found with the silica. It may reach one-third of the total amount.

If in correcting the silica, toward the end of the evaporation, when the hydrofluoric acid has been driven off and the sulphates begin to appear in solid form, the residue has a peculiar milky or enamel-like appearance, it may be taken as evidence of much phosphorus and titanium, though possibly the appearance may be due to zirconium with the other elements named. Although observed in but one series of rocks,<sup>a</sup> the appearance is so unusual and striking that it was deemed worthy of record.

It might be supposed that the residue would contain most of the barium of those rocks carrying that element together with sulphur or sulfates, but in my experience this is not so. Only when there is considerable excess of  $\text{SO}_3$  over  $\text{BaO}$  will any of the latter be present in the residue, and in the vast majority of cases there is none at all. This is due to the appreciable solubility of barium sulphate in hot hydro-chloric acid, coupled with the fact that barium is so minor a constituent of most rocks. Should some of it be present in the residue, its removal and estimation at this stage are not necessary, as it can be more conveniently recovered later, together with the silica accompanying the alumina, etc., precipitate (f.  $\beta$ , p. 90).

Quite as rarely is calcium or magnesium ever a component of the residue if the decomposition of the rock powder was complete at the outset. I have repeatedly proved that the residue, after re-solution, is quantitatively precipitable by ammonia in presence of an ammonium salt.

## f. PLATINUM IN FILTRATES.

The filtrates from the silica always contain notable amounts of platinum. This arises in very small degree from the crucible fusion, unless niter was added; in a larger degree indirectly from the action of hydro-chloric acid on the manganate, vanadate, and sometimes chromate of sodium; and, if much iron is present, in no small degree from the reduction of ferric chloride to ferrous by the platinum of the dish. This last reaction has attracted little attention until recently, but is

<sup>a</sup> See second footnote, p. 90.

mentioned by Gmelin-Kraut,<sup>a</sup> and can be readily demonstrated by evaporation of ferric chloride in platinum.

The removal of this platinum before precipitating alumina and iron is not necessary (but see third footnote, p. 90), and to do so involves the reoxidation of all iron and subsequent boiling to remove or destroy the excess of oxidizing agent, together with the expenditure of much valuable time. The iron is already oxidized by the fusion, and needs no further help in that direction. Nevertheless, if time is not a prime object, its removal by hydrogen sulphide is to be recommended. In the following descriptions, however, it is assumed that the platinum has not been gotten rid of at this stage.

#### 7. METALS PRECIPITABLE BY HYDROGEN SULPHIDE.

The presence in appreciable amounts of metals precipitable by hydrogen sulphide, except perhaps copper, is of so infrequent occurrence in most rocks that discussion is unnecessary in their connection. In case it is necessary to precipitate them at this stage, however, it is always well to bear in mind that a little titanium may be thrown down along with them. Separations of the silica should be made in porcelain, to eliminate platinum; or, better still, the quantitative determination of these metals should be made in a separate portion of the rock broken up by the action of hydrofluoric and sulphuric acids.

#### 8. ALUMINUM—TOTAL IRON.

##### A. INDIRECT METHOD FOR ALUMINUM. (TITANIUM.)

###### a. PRELIMINARY REMARKS.

The difficulties in the way of successfully producing and handling the complex precipitate which is obtained in the next stage of the analysis and which should contain the aluminum, iron, titanium, and phosphorus, besides chromium, vanadium, zirconium, and rare earths if present, and according to some preferences the manganese, are very great. It is fully recognized that there are objections to parts of the procedure that is usually followed in this laboratory, but the same may be said of any other known procedure, including that preferred by Dittrich. It is believed that the Survey method, which in its main features is closely modeled after old and well-known methods, will in competent hands give better results on the whole than any other.

The common practice in this laboratory is to find alumina by difference, after deducting from the precipitate produced by ammonia or

<sup>a</sup> Anorg. Chemie, 6th rev. ed., vol. 3, 1875, p. 359.

sodium acetate the sum of all other oxides this precipitate may contain. Of these, only ferric oxide, titanite oxide, and the trace of silica are determined in this portion (see also third footnote, p. 90), those of phosphorus, vanadium, chromium, zirconium, and sometimes manganese being looked for in other portions of the rock powder. This throws on the alumina all errors involved in their separate determinations; but these may balance, and in any case the probable error can hardly be as high as that involved in the direct weighing of the alumina itself, considering the difficulty of effecting a satisfactory separation of it from all the other admixtures, an operation which would, moreover, immoderately extend the time required for each analysis.

**b. PRECIPITATION OF ALUMINUM, IRON, ETC., WITHOUT PRECIPITATING MANGANESE.**

*α. Precipitation by ammonia.*—Two precipitations by ammonia at boiling heat are usually quite sufficient to separate iron, aluminum, phosphorus, vanadium, chromium, titanium, and zirconium, if all these are present, from nickel, manganese, the alkaline-earth metals, and magnesium, provided ammoniacal salts are present in sufficient quantity. This last point is of special importance as regards magnesium, and failure to observe it is doubtless the reason why many old analyses, and sometimes modern ones, show utterly improbable percentages of alumina, especially as chemists were formerly often satisfied with a single precipitation. The necessary ammonium chloride is better obtained by the use of purified ammonia water and hydrochloric acid than by the addition of the solid salt, which is seldom pure. The chemist should satisfy himself what amounts of ammonium salt are needed to hold in ammoniacal solution the maximum percentage of magnesium that the rock he is analyzing might contain. With extremely high magnesian rocks a third precipitation is generally requisite.

The precipitations are made in a bulk of 300 to 400 cm.<sup>3</sup>, at boiling heat, in a platinum dish if possible, otherwise in one of the borosilicate beakers of the Jena or Nonsol type. The ammonia should be as free from carbonate as possible, as well as from any nonvolatile matter, and it need not be added in any great excess. The complete boiling off of this excess is unnecessary, as pointed out by Genth and Penfield, since it is apparently the washing with pure water and not the free ammonia which carries small amounts of alumina into the filtrate. S. L. Penfield and D. N. Harper<sup>a</sup> recommend washing with a dilute solution of ammonium nitrate (20 cm.<sup>3</sup> nitric acid, neutralized by ammonia, to the liter), and also the solution of the first precipitate in nitric instead of hydrochloric acid, in order to shorten the washing.

<sup>a</sup> Am. Jour. Sci., 3d ser., vol. 32, 1886, p. 112.

there being no chloride to remove, both of which are excellent suggestions.<sup>a</sup>

For a reason mentioned under e (p. 87) the second precipitation may with advantage be made in the presence of macerated filter paper, as recommended by Dittrich.<sup>b</sup>

In the presence of little or no manganese, and also in the absence of iron, the ammonia method of precipitation is preferable by far to the basic-acetate method described in the following section, but it will occasionally happen that the separation from even very small amounts of manganese is altogether incomplete, and the uncertainty of insuring this separation led me to employ the basic-acetate method for the first precipitation in all cases where manganese is present—and the exceptions are few—even though the precipitation of alumina is sometimes less complete than by ammonia and in spite of other admitted defects, as, for instance, a tendency of the precipitate to run through the filter on washing.<sup>c</sup>

The most likely explanation of the persistent retention of small amounts of manganese by the precipitates produced by ammonia or acetates, even after repeated precipitation, seems to be that a portion of the manganese becomes oxidized to the  $Mn_2O_3$  state and remains so when redissolved, being then in a fit condition for reprecipitation.

*B. The basic-acetate precipitation.*—Although for this precipitation most writers seem to prescribe or prefer sodium acetate, except when the presence of a fixed alkali would interfere with subsequent operations, there does not seem to be any valid reason why ammonium acetate should not do quite as good service, besides having the advantage of easy removability afterwards in case of need. Also there is probably no good reason why the corresponding carbonates instead of the hydroxides should be used for neutralizing the hydrochloric solution before adding the acetate, other than the fact that they were, when the methods originated, and still are, easier to obtain comparatively free from silica and alumina. It is to be borne in mind that with precipitates low in iron and high in aluminum the separations are less satisfactory than when the reverse condition prevails. In rocks, alumina usually predominates, often largely, over iron oxide, so

<sup>a</sup> A single objection, and this of a practical nature, weighs against these suggestions. It will not do to evaporate to dryness filtrates containing both chlorides and nitrates in the same platinum dish, still less to drive off the mixed ammoniacal salts in platinum. With much iron present the final precipitation from a nitric-acid solution is a decided advantage, but is less so in the case of alumina free or nearly free from iron. Contrary to numerous statements and general belief, there is no danger of loss of aluminum chloride by volatilization when the precipitate is ignited before complete removal of ammonium chloride.

<sup>b</sup> Anleitung zur Gesteinsanalyse, 1905, pp. 10, 11, 14; Ber. Deutsch. chem. Gesell., vol. 37, p. 1840, 1904.

<sup>c</sup> The fact must not be overlooked that certain of the rare earths may pass completely into the filtrate if the basic-acetate method is followed. If then, later, on rendering the combined filtrates ammoniacal an unexpectedly large precipitate appears, this should be carefully examined as to its nature. In an analysis of piedmontite from Maryland over 2 per cent of rare earths, including cerium and others not identified, were quantitatively separated in this way from iron, alumina, etc.



that none of the methods of making the basic-acetate separation that involve the presence of notable quantities of free acetic acid are permissible, because of the solubility of aluminum hydroxide in this acid even when it is very dilute.

The cold and not excessively acid solution, not exceeding 100 cm.<sup>3</sup> in bulk and contained in a beaker of Jena or Nonsol glass, is first neutralized with great care by adding a freshly prepared solution of sodium carbonate, drop by drop. When the solution begins to deepen in color, the addition of the carbonate is continued with less speed and with longer stirring between the successive drops. When, now, the precipitate that each drop causes begins to dissolve very slowly, no further addition may be made till the liquid becomes quite clear again. With practice, it is not difficult to recognize this condition, even when the liquid is very dark. The intensity of the color will depend on the amount of iron present and the degree of dilution; therefore with small amounts of iron one is much more liable to overstep the end point than with solutions rich in iron. When, in spite of vigorous stirring, the turbidity after a final drop seems rather to increase than diminish, one or, if need be, two drops of acid are added. If this does not clear the liquid, it is best to add a slight excess and to repeat the neutralizing process.

Two or three grams of sodium acetate dissolved in a small amount of water are now poured in and then boiling water till the total bulk is about 400 cm.<sup>3</sup>. The beaker is placed over a lamp and its contents brought to boiling and kept in that state for two or three minutes. As soon as settling has taken place filtration is proceeded with. It is generally best to dispense with the pump at this stage and to use a filter of such size that the whole of the precipitate can be conveniently brought upon it without filling to the top. The washing is to be done with hot water, to which has been added a very little sodium acetate, for the purpose of guarding against an otherwise inevitable turbid filtrate.

The precipitate, having been sucked dry at the pump, is redissolved in hydrochloric acid, reprecipitated by ammonia as in *a*, washed, sucked dry, and treated as in *e* (p. 87), after recovery as in *d* (p. 86) of the portions of aluminum and iron that may have passed into the filtrates.<sup>a</sup>

<sup>a</sup> *Mittasch's method for the basic-acetate separation of manganese from iron.*—A study of the conditions necessary for successful separations of large or small quantities of manganese from iron by one treatment has been made by A. Mittasch (Zeitschr. anal. Chemie, vol. 42, 1903, p. 492), who gives the following directions for the use of ammonium acetate. It must be borne in mind, however, that this method is not applicable to aluminous materials.

The solution, containing not much free acid (hydrochloric) and preferably cool, whose volume should not exceed 100 cm.<sup>3</sup>, is neutralized in a beaker with concentrated ammonium-carbonate solution (20 grams per liter, about double molar) till the precipitate that is formed begins to redissolve slowly, then with weaker solution (about one-tenth molar) till a slight precipitate persists for one or two minutes in stirring. Next is added, according to the amount of this precipitate, 3 to 5 cm.<sup>3</sup> of acetic acid (double normal), if the commercial article of ammonium acetate, which nearly corresponds to the

## C. PRECIPITATION OF IRON, ALUMINUM, ETC., TOGETHER WITH MANGANESE.

*α. By ammonia and ammonium persulphate.*—This method has for its object the coprecipitation of manganese with a view to simplifying subsequent operations. It is, however, defective in case the rock carries appreciable amounts of barium and strontium or is very high in calcium, by reason of the certain partial precipitation of some of one or both of the first two as sulphates and its possibility in the case of calcium. It is probable that calcium can be completely held in the filtrates by two precipitations in analyzing almost any silicate rock, but this is not at all certain with respect to limestones, a point that has not yet been investigated. If neither strontium nor calcium is present in sufficient amount to be retained in the precipitate, there is no vital objection to the method on this score, for barium can be recovered, as in f. *β*, page 90.

The applicability of the method as above limited being assumed, it may be carried out as follows, though too few tests have thus far been made in this laboratory to warrant its unqualified recommendation.

To the filtrate from the silica contained in a platinum dish, and in bulk from 100 to 200 cm.<sup>3</sup>, is added enough hydrochloric acid to prevent precipitation of manganese when made ammoniacal (see b. *α*, p. 82). Heat is applied, and about 1 gram of ammonium persulphate<sup>a</sup> free from all interfering impurities is added. When the liquid

acid salt  $(\text{NH}_4)_2\text{C}_2\text{H}_3\text{O}_5$  is used, or 10 cm.<sup>3</sup> if the normal salt is used. This last is made by neutralizing acetic acid with ammonia. The solution is then diluted with water to about 400 cm.<sup>3</sup> and brought nearly to the boiling point. Any precipitate that may have appeared being disregarded, 20 cm.<sup>3</sup> of the acid acetate (one-half molar) or 5 cm.<sup>3</sup> of acetic acid and 10 cm.<sup>3</sup> of the normal acetate (molar) is added, and the boiling temperature is maintained for a minute longer. Precipitation is complete even at 60°, but filtration is easier if the boiling temperature is reached. As soon as the solution is settled filtration is begun and the washing performed with hot water at first, then with hot water containing some acetate and a little acetic acid. Any adhering film is removed from the glass with hydrochloric acid and reprecipitated with ammonia. If this is added to the main filter it must not be done till the large precipitate has been washed completely, because of the danger of precipitating, by the ammonia, manganese that has not yet been washed out.

*The Brunck-Funk separation of iron as basic acetate from manganese, zinc, nickel, and cobalt.*—The following method of O. Brunck (Chem. Zeit., vol. 28, 1904, p. 514), slightly modified and extended by W. Funk (Zeitschr. anal. Chemie, vol. 45, 1906, p. 181), is claimed to afford a complete separation by one treatment. Its applicability to aluminous materials has, however, not been tested, and it is quite improbable that it would work with rocks containing phosphorus and titanium.

The method differs from other modifications of the acetate process in the avoidance of the delicate neutralization, by removing excess of acid by evaporation and in the addition of potassium chloride to form a double salt with ferric iron, and thus prevent the separation of a basic chloride during the drying. As given by Funk for materials soluble in acids the procedure is as follows:

To the chloride solution in a large dish is added potassium chloride (about 0.35 gram to 0.1 gram of iron), the solution is evaporated to apparent dryness on the steam bath, but without expelling all of the free acid. The residue is broken up superficially and the drying on the bath continued for five to ten minutes. It is then taken up in 10 to 20 cm.<sup>3</sup> of cold water, with which it should give a clear solution. It is requisite that there shall remain a slight amount of free acid to set free acetic acid from the acetate in the next stage of the operations. One and a half to two times the theoretically needed quantity of sodium acetate (3 molecules of  $\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$  to 1 atom of iron) is now added, in a solution which has been rendered slightly acid with acetic acid if originally alkaline. After diluting with cold water (to 400 to 500 cm.<sup>3</sup> for 0.2 gram iron) the solution is gradually heated with stirring to the point of precipitation (60° to 70°), allowed to settle, decanted, collected on the filter, and washed with hot water.

<sup>a</sup>This reagent as sold must be purified. Jannasch (Prakt. Leitfaden d. Gewichtsanalyse, 2d ed., 1904, p. 179) accomplishes this as follows: To a saturated warm solution (not over 95°) ammonia is

begins to boil, ammonia is added carefully till in decided excess, as shown by the odor, and boiling is kept up for several minutes. The precipitate is allowed to settle and is quickly filtered and washed with hot water three or four times and sucked dry by the pump. It has not been determined if such a very dilute solution of persulphate has any appreciable effect on glass, but apparently not, to judge from my own limited experience. The precipitate is then dissolved in a small amount of hot hydrochloric acid containing a few drops of sulphurous acid, and after addition of well-macerated filter paper reprecipitated precisely as the first time. It may then be washed three or four times with hot water containing a few drops of ammonia, then ignited in the paper, and weighed as  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Mn}_2\text{O}_3$ ,  $\text{TiO}_2$ , and  $\text{P}_2\text{O}_5$ . Any chromium in the rock should be in the filtrates, wholly oxidized by the persulphate, but it will be recovered with the unprecipitated traces of alumina, as detailed under d.  $\gamma$ , page 87, and should be added to the main precipitate above. It is not best to attempt to determine the chromium colorimetrically in the filtrate.

*$\beta$ . By ammonia and ammonium sulphide.*—Although this old method of precipitation can be employed in special cases, it is not well adapted generally to silicate rocks, and will, therefore, not be described in this connection.

#### d. RECOVERY OF IRON AND ALUMINUM FROM THE FILTRATES.

*$\alpha$ . After an ammonia precipitation.*—The filtrates are evaporated, always in platinum if possible and allowable, to a small volume. If ammonium salts are present in abundance, it is advisable to evaporate the filtrates separately, the second to dryness. Its dry contents, almost wholly ammonium salt, are expelled by heat, the slight residue being taken up with hydrochloric acid and added, whether wholly soluble or not, to the other filtrate reduced in bulk. To this ammonia is now added and the evaporation continued, the solution being kept alkaline, in order to coagulate the small amount of alumina (and usually iron hydroxide) that will invariably be found. This is collected on a small filter and the filtrate caught in a flask of 150 to 200 cm.<sup>3</sup> capacity, if manganese is to be determined, otherwise in a platinum dish of suitable capacity for the calcium precipitation. The small precipitate is washed with hot water, burned, and added to the main one.

*$\beta$ . After a basic-acetate precipitation.*—The first filtrate is evaporated to, or nearly to, dryness, in order to get rid of the excess

added till its odor persists. [More will have to be added at intervals, since the continual decomposition of persulphate sets free sulphuric acid. —W. F. H.] When the precipitate of alumina, etc., has settled, the solution is filtered as rapidly as possible through asbestos, a porcelain funnel being used, since the strong solution attacks both paper and glass markedly. When cooled to about 30°, the clear filtrate is poured into twice its volume of alcohol and further cooled by ice applied externally. After half an hour the precipitated persulphate is collected on a filter, washed with alcohol, and then with ether, and dried in the air. The alcohol must be thoroughly removed. A commercial article purified thus by me (W. F. H.) was found to contain no interfering impurities, though not free from alkali.

acetic acid. The residue is then taken up, with only enough hot water to keep it in solution, and the dish is replaced on the bath for a short time. The precipitate, which will generally be appreciably larger than that obtained in  $\alpha$ , is collected on a small filter (7 cm.), and the filtrate in a flask of 150 to 200 cm.<sup>3</sup> capacity. The second or ammoniacal filtrate, having been separately evaporated to small bulk, with the addition of a few drops of ammonia toward the end, serves as first wash water for the other dish and for the precipitate on the filter. Both dishes and the filter are finally rinsed with hot water. The ammoniacal filtrate contains enough ammonium salts to prevent precipitation of magnesium in the first filtrate when the two come together.

If manganese peroxide has been deposited on the surface of the dish it is removed by hydrochloric acid and a drop or two of sulphurous acid, which mixture is then passed hot through the filter. A reprecipitation by ammonia is then made, and the precipitate collected again on the filter and added to the main one, the filtrate passing into the flask containing the previous filtrate. If much manganese is present, of course a second precipitation by ammonia of the small precipitate may be required. In these cases there is no difficulty in getting all the manganese into the filtrate.

*$\gamma$ . After precipitation by ammonia and ammonium persulphate.*—The filtrates are evaporated as in  $\alpha$ . The second is carried to dryness and the ammoniacal salts are removed by ignition. The residue is treated with a little hot hydrochloric acid and added to the first. A drop or two of sulphurous acid is then added if chromium is present, as will usually be shown by a yellowish color of the solution. The aluminum, chromium, and trace of iron are then precipitated by ammonia in slight excess and the digestion allowed to proceed on the bath till the slight precipitate has coagulated. This is collected on a small filter (7 cm.) and washed with hot water, the filtrate being collected as in  $\alpha$  and  $\beta$  if nickel or zinc are to be looked for, otherwise in a large platinum dish for treatment as in 10 (p. 101).

The precipitate is redissolved in hydrochloric acid, reprecipitated in small bulk of solution by ammonia, and finally added to the main precipitate obtained in c, page 85.

**e. IGNITION OF THE PRECIPITATE OF IRON, ALUMINUM, ETC., OXIDES.**

The combined precipitates of alumina, etc., obtained in either a, b, c, or d, are ignited without preliminary drying in the crucible containing the residue from the silica (6. B. c, p. 79), unless considerable iron is present. In that case the main precipitate is dried and removed as far as possible from the paper, which is ignited separately to prevent partial reduction of a portion of the iron oxide. This is especially necessary when the precipitates were obtained without

ing macerated filter paper, in which case they are, after ignition, mpy, and any magnetic oxide that may have been formed can not be wholly reoxidized by heating or by treatment with nitric acid (see 5. A, p. 59). They are also slow to yield to subsequent treatment. A precipitate formed in presence of thoroughly macerated paper, as recommended by Dittrich, on the other hand, yields on ignition a powder, and the fine state of division makes reoxidation of any reduced iron easy, besides wonderfully shortening the time required for subsequent re-solution.

Alumina in the quantities ordinarily found can not be fully dehydrated by the full heat of the Bunsen burner. It must be blasted for five or ten minutes. If iron is present in large amount this last operation must be conducted so as to insure access of air to the crucible (p. 73).

For ignitions of this kind, which involve no very prolonged heating, an electric furnace is admirably adapted by reason of its nonreducing atmosphere, provided the temperature is not high enough to cause by temperature alone reduction of ferric to magnetic oxide of iron.

The empty weight of the crucible, which may have changed since igniting the silica in it, is found after treatment of its contents as in the next section.

#### f. RECOVERY OF SILICA AND POSSIBLE BARIUM IN THE ALUMINA PRECIPITATE.

*α. Silica.*—The weighed precipitate is transferred as far as possible to another crucible of 25 to 30 grams weight. The small adhering residue, chiefly that originally derived from the silica, is brought into solution by fusion with a small amount of sodium or potassium pyrosulphate. This fusion takes but a few minutes, and must not be continued beyond the time actually needed, otherwise it will be impossible, after cleaning and igniting, to get the correct weight the crucible possessed after the ignition of the alumina.<sup>a</sup> The hot liquid is poured onto the main mass of precipitate in the other crucible, more pyrosulphate<sup>b</sup> is added (up to 7 grams in all) and the fusion is renewed and continued until the whole has been dissolved, which in the case of the potassium salt is always easy to be seen, even when the liquid appears dark red and opaque, by removing the crucible from the flame to a good light and allowing to cool. At one point in the cooling the liquid becomes transparent and allows the bottom of the crucible to be seen. This is less the case with the sodium salt.

<sup>a</sup> It will be remembered (see page 78, footnote c) that long blast or furnace ignition of the silica may increase the weight of the crucible by an appreciable amount, hence the need for getting its

<sup>b</sup> Sodium bisulphate for bringing this precipitate into solution

When all is dissolved<sup>a</sup> the melted mass is poured into a large, dry platinum dish, what adheres to the two crucibles is removed by diluted sulphuric acid, and the solution, with rinsings, is poured into a dish. Much more dilute sulphuric acid is added and the dish heated till the solid is dissolved. The solution is evaporated as far as can be done on the steam bath, then gradually heated higher till fumes of sulphuric acid come off copiously. During this heating the mass is apt to darken from reduction and separation of dissolved platinum. The acid should have been used in such quantity that after cooling the mass is pasty and not solid, for it then dissolves readily, on heating, the water, which is now to be added, the sodium salt dissolving much more readily than the potassium salt.

The dish is placed on the bath and soon the silica can be seen in agglutinated form at the bottom. It is collected on a small filter, well washed with hot water, burned, and weighed in a platinum crucible. Being rarely pure, it has to be corrected by a few drops of hydrochloric and a single drop of sulphuric acid, and reweighed. The slight residue in the crucible is brought into solution by pyrosulphate and added to the main portion, for the treatment of which see B, page 90, after filtering from barium if present (see  $\beta$ , p. 90).

The amount of silica thus recovered varies from 2 to 4 milligrams after two filtrations of the main silica, and is, of course, to be added to  $\alpha$ .

Only in this way is the full amount of the silica in the alumina precipitate to be recovered.<sup>b</sup> Formerly it was customary to regard that remaining undissolved when the pyrosulphate melt was taken up with

acid as applied to the bisulphate, but is swept away if the latter is first converted into pyrosulphate, which is always its condition after the frothing has ceased. To effect this change it is only necessary to melt a large amount of the salt in a platinum dish and keep it in fusion till spattering ceases and the fumes begin to come off freely. If the liquid is then poured out into other dishes it cools in thin sheets or cakes, which can be readily broken up and bottled for future use. With such material the action is always tranquil, and the action on the alumina, etc., much more intense and speedy than with ordinary bisulphate. If a good article of the latter is not to be had by purchase, it is only necessary to mix and melt together equivalent weights of sulphuric acid and pure normal sulphate until the fumes of the acid have been mostly expelled and the conversion to pyrosulphate accomplished. More lately, the recommendation of J. Lawrence Smith (*Am. Jour. Sci.*, 2d ser., vol. 40, 1865, p. 248) to use sodium instead of the potassium salt has been followed with satisfaction. This salt acts more readily and forms a more soluble double salt with aluminum, but has the slight disadvantage that progress of decomposition of the ignited mass is not so readily followed because of the greater tendency of the sodium salt to crust over.

Another objection is the action on the crucible of the fused pyrosulphate and the consequent necessity of removing the dissolved platinum. To obviate this and likewise to effect more speedy solution of oxides E. Deussen (*Zeitschr. anorg. Chemie.*, vol. 44, 1905, pp. 423-426), fuses the finely powdered oxides with acid potassium fluoride (about 1 gram). The mass melts at first over a low flame, then diffuses. Its solution is evaporated with sulphuric acid to expel most of the fluorine, then reduced with hydrogen sulphide or sulphurous acid and titrated for iron. The use of the fluoride is, however, incompatible with accurate work in silicate analysis, since the silica in the ignited oxides can not be determined. Moreover, the oxides must be finely powdered and consequently an aliquot part only can be taken for the fusion. The expulsion of fluorine by sulphuric acid must furthermore be thorough if titanium is to be determined in this solution.

Should a small portion of the precipitate have escaped solution no particular harm is done. The further treatment is not thereby changed.

For proof see *Jour. Am. Chem. Soc.*, vol. 24, 1902, pp. 368-370.

water or dilute acid as the total amount. It is, however, but a small portion of what the alumina held, the greater part having formed alkali silicate during the fusion and remaining in solution afterwards, unless set free and rendered insoluble by the treatment above given.

The silica thus recovered does not, however, represent quite all of that which originally escaped separation by evaporation with hydrochloric acid. A small part (roughly 1 to 2 milligrams for a single precipitation and a total of 1 centigram of silica in the experiments detailed elsewhere<sup>a</sup>) is not precipitated with the alumina and escapes recovery altogether. It is therefore apparent why a thorough separation of silica at the start is so urgently called for.

*β. Barium.*—Should by any chance the alumina precipitate have held barium sulphate, something which has not happened in my experience with rocks, unless ammonium persulphate was used as in c. *α*, page 85, it will be found as a contaminant of the silica recovered after the pyrosulphate fusion. The weighed residue remaining after expulsion of this silica by hydrofluoric acid is dissolved (except the platinum it may have held) by a little pyrosulphate and the cooled melt redissolved in dilute sulphuric acid. Any barium will then be left in the form of sulphate,<sup>b</sup> and can be separated by flotation from the heavier platinum.

## B. DETERMINATION OF IRON IN THE PRECIPITATE OF ALUMINA, ETC.

### a. WITHOUT REGARD TO THE PRESENCE OF VANADIUM.

*α. Reduction of the iron.*—Into the strongly acid and cool filtrate obtained in f. *α*, *β*, pp. 88–90, held in a beaker, is introduced hydrogen sulphide for the reduction of the iron to the ferrous state and the precipitation of the platinum resulting from the pyrosulphate fusion. This last effect is rendered complete by causing the solution to boil with continued passage of the gas, whereby the sulphur becomes coagulated and readily filterable. The gas current is not interrupted till

<sup>a</sup> Jour. Am. Chem. Soc., vol. 24, 1902, p. 369.

<sup>b</sup> Some years ago, in a series of analyses of rocks from the Leucite Hills, in Wyoming, there was obtained at this stage, when it was customary to dissolve the melt in cold water preliminary to precipitation of titanium by boiling the neutralized sulphuric solution in presence of sulphur dioxide, a white, more or less flocculent residue which amounted to 1 to 3 per cent of the rock and which was at first taken to be a mixture of tantalic and columbic acids. Eventually, it was found to consist apparently of nothing but  $\text{TiO}_2$  and  $\text{P}_2\text{O}_5$ , with perhaps a little  $\text{ZrO}_2$ . By repeated fusion with acid potassium sulphate and leaching with cold water it could be gradually brought into solution. It was these rocks which furnished the most striking instance of the peculiar milky sulphate residues mentioned on p. 80 as derived from the ignited silica.

Knop (Zeitschr. Kryst., vol. 10, 1885, p. 73) seems to have obtained a similar mixture in analysing minerals from the Kaiserstuhl in Baden, but its nature was not ascertained, though it was suspected to be, if not silica, columbiferous titanate acid.

<sup>c</sup> It may be mentioned that the precipitation of platinum from a hot sulphate solution is far quicker and cleaner than from hydrochloric acid. Further, this platinum sulphide, when ignited in the crucible in which the bisulphate fusion was made, should weigh together with the crucible itself what the latter weighed before the main silica precipitate was ignited in it; in other words, the weight of the platinum recovered by hydrogen sulphide should equal the loss in weight of the crucible due to attack by the bisulphate. In somewhat rare instances this will not be so, but the weight will be greater, showing

the solution has cooled somewhat. This is then filtered <sup>a</sup> warm into a half-liter flask, hydrogen sulphide is again introduced for a few minutes, with production of slight milkiness if any iron had become oxidized during filtration, the flask is then connected with a carbon-dioxide generator already in action for a time, and the solution brought to boiling to expel the hydrogen sulphide. The expulsion is aided by a rapid current of carbon dioxide,<sup>b</sup> the end point being found by occasional testing of the escaping gas with lead-acetate paper.<sup>c</sup> When this point is reached the flame is removed and the flask allowed to cool without interrupting the gas current. The cooling is hastened by placing the flask in a basin of cold water.

*β. Titration of the iron.*—A burette with permanganate solution having been made ready, the gas tube is rinsed into the flask and the permanganate at once run into the latter without further dilution of the solution, which is kept in rotatory agitation by an appropriate movement of one hand. The bulk of the solution will be from 100 to 200 cm<sup>3</sup>. The drops of the titrating fluid that may fall upon the neck of the flask are washed down by aid of a wash bottle. The strength of the permanganate solution would best not much exceed

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gain in platinum which may amount to a milligram. Tests have shown that this is not due to retention of platinum by the main  $\text{Al}_2\text{O}_3$ , etc., precipitate; hence, it must come from platinum mechanically loosened from the dish during the drying and powdering of the silica preparatory to its collection on the filter, or to some insoluble compound of platinum formed during evaporation and drying of the silica. It may also be in part or wholly due to contamination from reduction of platinum during evaporation of the filtrate from the basic-acetate separation. It will be remembered that from this filtrate a small amount of iron and alumina is recovered and added to the main precipitate. Hence it is always well in fine work to collect the sulphide and weigh the platinum in the original crucible, deducting any excess from the alumina, or else to get rid of the platinum by hydrogen sulphide before proceeding to the precipitation of alumina, etc. (See 7, p. 81.)

<sup>a</sup> Filtration is not necessary if only precipitated sulphur and no sulphides are in suspension, since this is without reducing action on cold permanganate solution, as H. A. Wells and W. L. Mitchell, and others before them, have pointed out. The above authors used this method of reducing ferric iron in titanite iron ores. (Jour. Am. Chem. Soc., vol. 17, 1895, p. 78; Chem. News, vol. 73, 1896, p. 123.) The observation of A. Coppadoro (Gazz. chim. ital., vol. 31, pt. 2, 1901, p. 217) that the precipitate of sulfur caused by reducing ferric solutions with hydrogen sulphide carries some iron as sulphide, even in strongly acid solutions and after thorough washing, need not be regarded. He seems to have operated on very concentrated solutions as compared with those here in question.

<sup>b</sup> The gas generated from every new lot of marble must be carefully tested for hydrogen sulphide. Even pure white marble is frequently not free from soluble sulphides—probably pyrrhotite, sphalerite, or galena—and the amount may be enough to appreciably affect the result of titration for iron. A good precaution to take is to wash the gas by passing it through a large U tube containing glass beads and a solution of copper sulphate.

<sup>c</sup> The test is most delicate when the flask is provided with a two-hole stopper fitted with inlet and outlet tubes of glass. If the outside opening of the outlet tube is small the concentration of the escaping steam and gas contributes to the sharpness of the test. Because of the possibility of the introduction of organic matter from the stopper by the action of the hot steam, it is safer to employ a cap of platinum foil with two holes, one for the gas entry tube, the other for its escape in a concentrated stream.

If the solution was free from milkiness at the start the operation of expelling hydrogen sulphide need not take more than ten to twenty minutes; otherwise a longer time will be required to remove the last traces. The immediate effect of boiling on any moderate amount of suspended sulphur is to coagulate it and clear up the solution completely. Some of the sulphur volatilizes with the steam, but a further portion appears to be converted by it to hydrogen sulphide, so that a faint reaction for that gas can be obtained as long as any sulphur remains. This is probably the explanation for Washington's direction (Manual of the Chemical Analysis of Rocks, 1901, p. 112) to boil for about two hours, since by his method of reducing the iron, without coagulating the sulphur, the filtrates are always strongly milky.



0.0025 gram of iron to the cubic centimeter, for the amounts of iron to be determined are moderate and often very small.

The results are strictly accurate, with the limitations set forth in the paragraph below, when care is taken with the reduction by hydrogen sulphide. The method is altogether superior to that involving the use of zinc, since no foreign impurity affecting the result is introduced, and the ever present titanium is not affected, nor is vanadium reduced below the condition of  $V_2O_4$ , whereas nascent hydrogen converts it, in part at least, to  $V_2O_3$ .

*γ. Further treatment of the solution after titration.*—The titrated solution is returned to a dish, which may be of porcelain, and evaporated to a bulk of much less than 100 cm.<sup>3</sup> if the rock is supposed to contain less than 1 per cent of titanium. For each additional 1 per cent of titanium the final bulk may be larger by 100 cm.<sup>3</sup>. The solution is transferred to a graduated flask with a capacity of 50, 100, or 200 cm.<sup>3</sup> or more, as the case may be, enough hydrogen peroxide free from fluorides is added to fully peroxidize the titanium, the flask is filled to the mark and well shaken, and the titanium is determined colorimetrically (see p. 110).

#### B. HAVING REGARD TO THE PRESENCE OF VANADIUM.

If vanadium is present the value found for iron will be in error by the amount of permanganate required to oxidize  $V_2O_4$  to  $V_2O_5$ . The amount of the correction will differ according as titration of the iron is made after reduction by hydrogen sulphide or nascent hydrogen. If the former is used, as should always be the case, because of the ever-present titanium, the vanadium is reduced by it to  $V_2O_4$ , which in its action on permanganate is equivalent to two molecules of FeO, while the reduction goes further with hydrogen. After the first transient pink blush throughout the liquid, the more slowly acting vanadium may require the addition of a drop or two more of permanganate before comparatively permanent coloration appears.

When the amount of vanadium in the rock is known a correction can be applied on the assumption that practically all the vanadium here collected, a point that needs further investigation. Various authors assert its precipitability with alumina and iron by ammonia and ammonium acetate, though Carnot<sup>a</sup> states that repeated precipitation by ammonia, ammonium carbonate, or ammonium sulphhydrate separates it from iron. My experience with ores very rich in vanadium shows that precipitation along with iron and aluminum is only partial. Ridsdale<sup>b</sup> has determined its precipitability with various metals and gives numerous figures which show an approximation to 90 per cent thus thrown down under the conditions prevailing in analysis of

<sup>a</sup> Comptes rendus, vol. 104, 1887, p. 1803; Zeitschr. anal. Chemie., vol. 32, 1893, p. 223.

<sup>b</sup> Jour. Soc. Chem. Industry, vol. 7, 1888, p. 73.

iron slags, the remainder passing into the filtrates and appearing in small part with the lime and to a greater extent with the magnesium phosphate. For all practical purposes it is probably safe to assume that the small amounts of vanadium met with in rocks are wholly in the alumina precipitate.

If the amount of vanadium in the rock is not known and great accuracy is necessary, caution requires the determination of the total iron to be made either in a separate portion or after reprecipitation from the above solution, as follows: Fuse with sodium carbonate, extract with water, bring the insoluble residue into sulphuric solution, reduce, and titrate as above directed. But unless a certain precaution is here observed an error greater than that which it is designed to avoid will be committed. Contrary to general belief the aqueous extract from the sodium-carbonate fusion carries a small but appreciable fraction of a per cent of iron, as I have repeatedly found by actual test. This iron is thrown out with the alumina (and silica, if present) by the usual methods of neutralizing the alkaline solution, and can be brought to light when the precipitate thus formed is treated with a fixed caustic alkali, or again fused with sodium carbonate and leached with water, when it remains wholly or in part undissolved. Hence it is necessary to collect this iron and add it to the main portion before titration.

#### C. DETERMINATION OF THE TRUE VALUE FOR FERRIC IRON.

The total iron in the rock having in one way or another been found, it remains to deduct an amount equivalent to the ferrous oxide the rock contains and a further amount corresponding to the sulphides often present, in order to get what may pass for the true value for ferric iron. That this is often only an approximation appears from the difficulties due to the presence of vanadium and the generally indeterminate effect of sulphides on the ferrous-oxide determination. (See pp. 138-140.)

#### C. METHODS AIMING AT THE MORE OR LESS DIRECT DETERMINATION OF ALUMINUM.

##### a. AFTER FIRST REMOVING IRON AS SULPHIDE.

Should it be desirable for any reason to effect an actual separation of aluminum, this may best be done, up to a certain point, after the pyrosulphate fusion (f, p. 88), by removal of the iron<sup>a</sup> by ammonium sulphide in ammonium-tartrate solution, evaporation of the filtrate, ignition of the residue with sodium carbonate and nitrate, and extraction with water, whereby titanium and zirconium are left on the filter

<sup>a</sup> This being first reduced to the ferrous condition by hydrogen sulphide in acid solution in order to obviate the possibility of precipitating some titanium, which otherwise is likely to happen. (Cathrein, *Zeitschr. Kryst.*, vol. 6, 1882, p. 246, and vol. 7, 1883, p. 250.)

as sodium salts while chromium and vanadium are carried into the filtrate as chromate and vanadate along with aluminum and phosphorus. The further separation of the last two from the chromium and vanadium is outlined under "Phosphorus" (B, p. 122). This is as far as the separation can well be carried, and the  $\text{Al}_2\text{O}_3$  must still be found by subtracting the  $\text{P}_2\text{O}_5$  from the combined weights of the  $\text{Al}_2\text{O}_3$  and  $\text{P}_2\text{O}_5$ . The possibility of loss of some  $\text{P}_2\text{O}_5$  by volatilization<sup>a</sup> during the bisulphate fusion must be borne in mind here, for if it takes place the final weight of  $\text{Al}_2\text{O}_3 + \text{P}_2\text{O}_5$  will not contain all the  $\text{P}_2\text{O}_5$ .

Some writers have recommended dissolving the ignited alumina, iron oxide, etc., in hydrochloric acid, but when the precipitate has been heated over the blast, as it should be, this is very ineffective.

#### b. BY EXTRACTION WITH A FIXED CAUSTIC ALKALI.

A favorite practice in some countries of Europe has been to fuse the ignited precipitate containing  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{P}_2\text{O}_5$ , etc.—or that of the  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{P}_2\text{O}_5$ , etc., after separation of iron by ammonium sulphide in tartrate solution—with sodium hydroxide in a silver crucible, or to boil the freshly precipitated mixture with a solution of the alkali, on the assumption that the oxide of titanium is hereby rendered wholly insoluble and thus separated from the alumina. This, however, is in part an error long since pointed out by F. A. Gooch,<sup>b</sup> who showed that pure titanite oxide is markedly soluble under both conditions of treatment. Experiments made by myself to test the extent of this error brought out the following interesting results:

When 0.045 gram of titanite oxide was fused by itself with sodium hydroxide, the clear aqueous extract of the fusion held 0.0031  $\text{TiO}_2$ , or about 7 per cent, determined colorimetrically. When freshly precipitated and boiled with the alkali the solubility was less. When fused with sodium carbonate but an infinitesimal trace was dissolved, which required strong concentration for its detection. When mixed with a large excess of alumina and fused with the caustic alkali, the solubility was still very marked, though less than when alumina was absent. With a large excess of ferric oxide, with or without alumina, no titanium could be detected in the unconcentrated filtrate.

It thus appears that fusion with caustic alkali after first removing iron involves an error in the gravimetric determination of both aluminum and titanium which does not appear if the iron has not been removed.

<sup>a</sup> H. Rose speaks of such loss when volatilizing sulphuric acid in presence of phosphoric acid (Handb. f. quant. Anal., Finkener ed., vol. 2, 1871, p. 575, and elsewhere), and the fact of such loss to a very marked extent has been confirmed in this laboratory.

<sup>b</sup> Proc. Am. Acad. Arts Sci., vol. 12, 1885, p. 436; Bull. U. S. Geol. Survey No. 27, 1886, pp. 16, 17.

## C. COMBINATION METHOD AFTER DITTRICH.

Evidently with a view to eliminating the sources of error inherent in the methods outlined in a and b above, Doctor Dittrich has evolved a procedure which is a combination of both and includes methods of determining titanium and zirconium. Since it involves considerably more work than that which has just been described in detail and has few compensating advantages beyond the colorimetric determination of titanium in absence of iron, it will be sufficient to refer the reader to it.<sup>a</sup>

## D. DIRECT PRECIPITATION OF ALUMINUM BY PHENYLHYDRAZINE.

A recent and promising method for the "direct determination of alumina in presence of iron, manganese, calcium, and magnesium" is that of Hess and Campbell,<sup>b</sup> but, as with the methods just considered, it involves finally weighing aluminum, phosphorus, and titanium together. Precipitation is made by phenylhydrazine, after first neutralizing the (preferably chloride) solution by ammonia and reducing iron by a saturated solution of ammonium bisulphite. Phenylhydrazine "precipitates aluminum from its solutions quantitatively as the hydroxide without a trace of the precipitate being redissolved in excess of the precipitant."

Dr. E. T. Allen has investigated the method in this laboratory<sup>c</sup> and confirmed the above authors' statements in the main, finding, however, that two precipitations are needed to free the precipitate entirely from iron. "The method is excellently adapted to the separation of very small quantities of aluminum, such as a milligram or even less, from a large excess of iron, a point of considerable practical importance."

Although such conditions will not be met with in ordinary rock analysis, it will not be out of place here to give the treatment as slightly modified by Doctor Allen:

The volume of the solution may vary, according to the quantity of alumina to be precipitated, from 100 to 200 cm.<sup>3</sup>. It should be heated and reduced by adding saturated ammonium bisulphite. From 5 to 20 drops, according to the quantity of iron, may be used. If the solution turns deep red (ferric sulphite), it is not acid enough, and a few drops of hydrochloric acid should be added, for the sulphite itself does not reduce ferric salts, at least not with rapidity. Now quickly bring to neutrality with ammonia, and then add several drops of dilute hydrochloric acid. If this last operation is done too slowly the oxygen of the air helps to form a little ferric hydroxide which does not always readily dissolve in the dilute acid. Finally, add from 1 to 3 cm.<sup>3</sup> of phenylhydrazine,<sup>d</sup> according to the weight of the alumina to be precipitated. If too little has been used, a few drops added to the filtrate will disclose the mistake. Stir until the precipitate has

<sup>a</sup> Dittrich, M., *Anleitung zur Gesteinsanalyse*, 1905, pp. 9-31.

<sup>b</sup> Jour. Am. Chem. Soc., vol. 21, 1899, p. 776; Chem. News, vol. 81, 1900, p. 158.

<sup>c</sup> Jour. Am. Chem. Soc., vol. 25, 1903, p. 421.

<sup>d</sup> The reagent should, of course, be free from inorganic impurities which could disturb the results. The author [Allen] found one sample which after persistently giving high results was proved to contain tin, which had probably been used in its preparation.

become sufficiently flaky and allow to settle. The supernatant liquid will now be plainly acid to litmus. One need not be disturbed if the precipitate has a brownish color, for it is not due to ferric hydroxide, but to the coloring matter contained by all phenylhydrazine which has not been freshly distilled. When the determinations are allowed to stand too long the air increases this oxidation product, and a brown insoluble scum forms on the surface of the liquid and on the sides of the vessel, which is rather troublesome to the analyst. • Fortunately equilibrium appears to be established in a short time. The vessels need not stand more than an hour, at any rate. The precipitate is washed by a solution of phenylhydrazine sulphite made by adding cold saturated sulphurous acid to a little phenylhydrazine until the crystalline sulphite first formed dissolves in the excess. The solution has an acid reaction. Five to 10 cm.<sup>3</sup> of this are used in 100 cm.<sup>3</sup> of hot water.

## 9. MANGANESE, NICKEL, COBALT, COPPER, AND ZINC.

### A. DIFFICULTIES IN THE WAY OF A CORRECT GRAVIMETRIC DETERMINATION OF MANGANESE.

The gravimetric determination of manganese in small amounts seems to be more of a stumbling block to the average chemist than that of almost any other of the frequently occurring elements met with in mineral analysis. This is due almost always to incomplete prior separation of elements which later suffer coprecipitation with the manganese. The error is therefore generally a plus one, and often amounts to many times the weight of the manganese actually present. The importance of thorough separation of the constituents treated in the foregoing pages is therefore manifest, particularly since to the inexperienced analyst very small amounts of manganese precipitated in the form of a higher hydroxide may easily mask a good deal of alumina or other colorless precipitates.<sup>a</sup>

<sup>a</sup> It is for these reasons that Washington deems it better for the novice in rock analysis to make no attempt to determine the manganese, but to allow the error from this neglect to distribute itself over the alumina, lime, and magnesia. The relative order of this distribution has been determined by George Steiger in the Survey laboratory recently, while analyzing a series of carbonate rocks, some of them highly siliceous. The results are instructive. Double precipitations were the rule, and iron and aluminum were thrown out by ammonia.

*Tests showing distribution of manganese in gravimetric separations.*

No.	Partial composition of rock.			MnO by colorimetry.			
	Al <sub>2</sub> O <sub>3</sub> . Fe <sub>2</sub> O <sub>3</sub> .	CaO.	MgO.	Total.	With— Al <sub>2</sub> O <sub>3</sub> . Fe <sub>2</sub> O <sub>3</sub> (by difference).	CaO.	Mg <sub>2</sub> F <sub>2</sub> O.
957	2.03	10.60	6.30	0.193	0.085	0.011	0.097
973	9.35	11.84	2.81	.311	.036	.023	.252
974	1.80	50.51	1.04	.700	.301	.087	.312
975	12.71	11.98	4.30	.442	.088	.016	.358
1126	.58	30.54	20.41	.281	.030	.030	.221
1128	.98	29.69	19.07	.245	.019	.055	.171
1130	3.49	3.99	.92	.016	.016	None	None
1131	1.00	28.04	19.11	.574	.032	.101	.441

The results in 974 and 1130 particularly show how the manganese once precipitated tends to remain with the alumina, presumably by reason of its having become peroxidized.

If the directions already given have been carefully followed, however, there will be little chance of error due to foreign contamination, either by alumina or magnesia, a sufficiency of ammonium salts being a guaranty against the latter. Regard must be had, however, to the rather remote possibility of the presence of rare earths which were not thrown out by the basic-acetate precipitation (see third footnote, p. 83), for they will appear at this stage.

## B. PRECIPITATION OF THE GROUP AND SEPARATION OF ITS CONSTITUENTS.

### a. THE AMMONIUM-SULPHIDE METHOD.

*α. Its advantages and disadvantages.*—On the other hand the usual methods of separation of manganese from the alkaline earths and magnesia by bromine or ammonium sulphide are imperfect, in part by reason of incompleteness, in part because of coprecipitation of small amounts of these metals. The former error, tho absolutely slight, is in rock analysis probably of more consequence than the latter. In regard to completeness of precipitation bromine offers no advantage over ammonium sulphide, and the latter has the advantage that by a single operation nickel, cobalt, copper, and zinc, if present, are likewise separated from the earth metals and alkalis. There need be no fear of overlooking nickel or copper, for under the conditions of the precipitation they are not retained in solution. It is for this reason chiefly that the ammonium-sulphide method is to be preferred. Most of the small amount of manganese that escapes precipitation is weighed later with the magnesium as pyrophosphate and can be readily corrected for by the colorimetric method described on page 99.

The precipitation of manganese in alkaline solution by hydrogen peroxide, as proposed by P. Jannasch and E. v. Cloedt,<sup>a</sup> a method which appeared to be simple and accurate, besides affording a separation from zinc, has been shown by C. Friedheim and E. Brühl<sup>b</sup> to be valueless.

*β. Precipitation by ammonium sulphide and separation of manganese and zinc from nickel, cobalt, and copper.*—Two or three cm.<sup>3</sup> of ammonia are added to the flask containing manganese, the earth metals, etc. (8. A. d. *α*, *β*, pp. 85, 86), and hydrogen sulphide gas is introduced to saturation, whereby manganese, nickel, cobalt, copper, zinc, and a small part of the platinum from the dish are precipitated. A second like amount of ammonia is now added. The flask, filled to the neck and corked, is set aside for at least twelve hours, and prefer-

<sup>a</sup> *Zeitschr. anorg. Chemie*, vol. 10, 1905, p. 405.

<sup>b</sup> *Zeitschr. anal. Chemie*, vol. 38, 1900, p. 681.

ably twenty-four, or even longer. The precipitate, collected and washed on a small filter with water containing ammonium chloride and sulphide, is extracted by hydrogen-sulphide water acidified with one-fifth its volume of hydrochloric acid (sp. gr. 1.11), manganese and zinc, if present, going into solution.

(For treatment of the ammonium-sulphide filtrate see 10, p. 101.)

#### b. MANGANESE AND ZINC.

The hydrochloric-acid filtrate is evaporated to dryness, ammonium salts are destroyed by evaporation with a few drops of sodium-carbonate solution, hydrochloric acid and a drop of sulphurous acid are added to decompose excess of carbonate and to dissolve precipitated manganese, and the latter is reprecipitated at boiling heat by sodium carbonate after evaporation of the hydrochloric acid. If zinc is present, it can be separated from the manganese after weighing. For the small quantities of manganese usually found the sodium-carbonate method of precipitation is to be preferred to that by bromine or sodium phosphate, as equally accurate and a time saver.

The manganese is weighed as  $Mn_2O_3$  and calculated to  $MnO$ , or, if present in some quantity, it may preferably be weighed as the sulphate,<sup>a</sup> or, finally, it may be determined colorimetrically with much greater certainty, as in C, page 99, when the amount is within the usual limits for rocks. In the last case, in the absence of zinc, the hydrochloric-acid solution of the sulphide may be used for the colorimetric test, after first evaporating off the hydrochloric acid with nitric or sulphuric acid.

#### c. NICKEL, COBALT, COPPER.

The paper containing nickel, cobalt, and copper is incinerated in porcelain, dissolved in a few drops of aqua regia, and evaporated with hydrochloric acid; the copper and platinum are thrown out warm by hydrogen sulphide, and in the filtrate made ammoniacal nickel and cobalt are thrown down by hydrogen sulphide. This liquid is then rendered faintly acid by acetic acid and allowed to stand. The sulphide of nickel after filtering is simply burned and weighed as oxide—its weight being always very small, hardly ever over 0.3 or 0.4 milligram—and is then tested for cobalt in the borax bead. The color of the last drop or two of the hydrochloric-acid solution, after decomposing the aqua regia, is a good indication of the presence or absence of nickel and cobalt. It is always well at this point to separate by ammonia any traces of iron that may be present, then to acidify and treat with hydrogen sulphide as above.

<sup>a</sup> Volhard, J., *Ann. Chem.*, vol. 198, 1879, p. 329. Gooch, F. A., and Austin, Martha, *Am. Jour. Sci.*, 4th ser., vol. 5, 1898, p. 209; *Zeitschr. anorg. Chemie*, vol. 17, 1898, p. 264.

It is somewhat unsafe to consider traces of copper found at this stage to belong to the rock if the evaporations have been conducted, as is usually the case, on a copper water or steam bath, or if water has been used which has been boiled in a copper kettle, even if tinned inside. Therefore, and because of its contamination by a little platinum, it is better to determine copper in a separate portion if its presence is indicated with certainty. (See 7, p. 81.)

### C. COLORIMETRIC DETERMINATION OF MANGANESE.

#### a. PRELIMINARY TREATMENT.

As has been shown above (p. 96), the gravimetric determination of manganese is subject to grave error, even when great care is used. This is due to a variety of reasons, such as incompleteness of separation from aluminum and iron, incomplete precipitation by ammonium sulphide, and contamination by other bodies. Because of the small amounts in question these errors may be relatively enormous, and they can not be depended on to balance each other. Therefore, it would be an advance if the total manganese could be determined in a separate portion of the sample, and in the main portion only that part which is weighed with the magnesium pyrophosphate, and this only as a correction to the magnesia. This course is readily feasible with carbonate rocks, which can usually be brought into a fit state in a few minutes without a fusion or the separation of silica. With silicate rocks more labor is involved, but the determination itself is very accurately performed by colorimetry, as in the case of carbonates.

The preliminary treatment consists in decomposing a gram or half a gram of the rock powder in a small platinum dish or capacious crucible (placed in the radiator shown in fig. 2, p. 29) with hydrofluoric and sulphuric acids till all is disintegrated. The hydrofluoric acid is then to be driven off by repeated evaporations with small portions of sulphuric acid, after which nitric acid free from chlorine is added, together with water, and as much of the residue is brought into solution as possible. What remains insoluble is usually barium and calcium sulphates. This is filtered off on a small filter, the filtrate is caught in a small beaker or flask, and the manganese determined as in the following paragraphs.

#### b. COLORIMETRIC DETERMINATION OF MANGANESE BY AMMONIUM PERSULPHATE.<sup>a</sup>

This method has the advantage over that by lead peroxide in that, unless chlorides happen to be in the solution, no filtration is necessary. Two solutions are needed, one of silver nitrate containing 2 grams of the salt to the liter, and one of manganous sulphate or nitrate

<sup>a</sup> Walters, H. E., Chem. News, vol. 84, 1901, p. 239; Proc. Eng. Soc. West. Pa., vol. 17, 1901, p. 257.



containing the equivalent of 2 milligrams of  $\text{MnO}$  in 10 cm.<sup>3</sup>. This last can be conveniently made by acidifying a standard permanganate solution, reducing it by sulphurous acid, and diluting appropriately. A solution of permanganate can be used directly as a color standard when very small quantities of manganese are in question. Ammonium persulphate in the solid form is also needed. The commercial article needs no purification ordinarily in this case.

The rock solution must be strongly acid with nitric or sulphuric acid and considerably less than 100 cm.<sup>3</sup> in bulk unless the manganese exceeds 1 milligram in weight. To it is added 10 cm.<sup>3</sup> of the silver solution for every milligram of metallic manganese, and it is transferred to a graduated flask of size suited to the solution. Should it show a turbidity from precipitated silver chloride, the liquid must be agitated and then filtered into the flask. There is now added about 1 gram of solid ammonium persulphate and the flask is placed on the steam bath or hot plate. Very soon the pink color of permanganic acid begins to appear and rapidly increases to a maximum. The flask may be removed soon after the color begins to show and placed in cold water when it is fully developed. If the depth of color is very great the solution must be poured into a larger flask. The flask is filled with water to the mark and the contents thoroughly mixed.

According to the depth of color there is now placed in another flask of 100 cm.<sup>3</sup> or less volume 10 cm.<sup>3</sup>, or some multiple of that amount, of the manganese solution, so that when both solutions are oxidized and the flasks filled the color in the standard shall be more intense than in the test solution. This solution is oxidized by the aid of silver salt and persulphate in the same manner as the other, cooled, filled to the mark, shaken, and poured into a burette. Ten cm.<sup>3</sup> is drawn off into one of the comparator cylinders shown in fig. 5 (p. 32), and diluted with water from a burette till the color equals that of the properly diluted and mixed test solution, a part or all of which has been poured into the companion cylinder. The observations are made exactly as given on page 32. If the form of colorimeter depicted in fig. 6 is used, the dilution of the standard solution is, of course, unnecessary. Nessler cylinders can be used instead of either of the colorimeters described. The results are very exact.

Should, perchance, a brown precipitate (due to deficiency of silver salt) have formed during the oxidation of the standard, it is best to start with a fresh portion, but in case this happens with the test solution the precipitate must be brought into solution by a little sulphurous acid, more silver salt added, and the oxidation repeated, if needed, after adding more persulphate.

## 10. CALCIUM AND STRONTIUM (BARIUM).

## A. SEPARATION FROM MAGNESIUM.

## a. PRECIPITATION AND IGNITION OF CALCIUM AND STRONTIUM OXALATES TOGETHER.

The platinum derived from the dish in the silica evaporation, except for the small portion precipitated with the manganese sulphide, is now wholly in the filtrate from the latter (9. B. a.  $\beta$ , p. 97). Its separation at this or any other stage is quite unnecessary; nor is the removal of ammonium chloride usually demanded, since there is no undue amount present in most cases, the first precipitation of alumina, etc., having been by ammonium or sodium acetate.<sup>a</sup> Therefore, without destroying ammonium sulphide the calcium and strontium are thrown out by ammonium oxalate at boiling heat, the precipitate, often darkened by deposited platinum sulphide, is ignited and redissolved in hydrochloric acid, boiled with ammonia to throw out traces of alumina sometimes present, and reprecipitated as before, but in a small bulk of solution. It is weighed as oxide, transferred to a small flask of 20 c.c. capacity, dissolved in nitric acid, evaporated to dryness at 150° or 160°, and the separation of strontium from calcium effected by ether-alcohol<sup>b</sup> as described below (B, p. 102).

The weight of strontia found deducted from that of the two oxides gives that of the lime. For treatment of the filtrates see "Magnesium" (11, p. 105).

## b. NOTES ON DOUBLE PRECIPITATION, WASHING, AND IGNITION OF THE OXALATES.

It may be said with regard to the separation of calcium from magnesium that two precipitations by ammonium oxalate are essential to attainment of correct results, not only for the complete removal of magnesium but of sodium as well, the retention of compounds of the latter element by calcium oxalate being now generally known. This effect has been thoroughly studied by T. W. Richards, C. T. McCaffrey, H. Bisbee.<sup>c</sup> They also call attention to the danger of too long boiling with hot water because of the very appreciable solubility of calcium oxalate. This is a point that needs greater attention than ordinarily receives from analysts.

Ignition is started with the precipitate moist. After charring and burning off the paper the crucible is to be covered and blasted in an inclined flame. It is a very mistaken idea that with a really fast it takes long to reach constant weight. Five minutes is fully ample for any amount of lime that is likely to be met

<sup>a</sup> If three precipitations by ammonia alone are depended on, the second and third filtrates are rapidly to dryness and the ammonium salts removed by ignition.

<sup>b</sup> *Zeitschr. anal. Chemie*, vol. 32, 1893, pp. 199, 312, for the latest improvements in this their application to considerable quantities.

<sup>c</sup> *Acad. Arts Sci.*, vol. 36, 1901, p. 375; *Zeitschr. anorg. Chemie*, vol. 28, 1901, p. 71.

with. For ordinary amounts the heat of a large and good Bunsen burner is in reality sufficient, since I was able by it to reduce 3 grams of calcium carbonate in fifty minutes to the oxide, or so nearly so that the blast afterwards caused a further loss of only 1 milligram. This, of course, was with the crucible covered. The lime should not be left over half an hour in the desiccator before weighing, for it gains weight appreciably if left long.

## B. SEPARATION OF STRONTIUM (BARIUM) FROM CALCIUM.

### a. BY ETHER-ALCOHOL.

The thoroughly dried nitrates obtained in A. a are treated with as little (rarely over 2 cm.<sup>3</sup>) of a mixture in equal parts of absolute alcohol and ether as may be needed to dissolve the calcium salt, solution being hastened by occasional gentle agitation. After standing overnight in the corked flask the insoluble matter is collected on the smallest possible filter and washed with more of the above mixture of alcohol and ether. After drying, a few cubic centimeters of hot water are passed through the filter, on which may remain a few tenths of a milligram of residue, which does not usually contain any lime or other alkaline earth and whose weight is therefore to be deducted from that of the lime, unless it can be shown that it is derived from the glass of the little flask in which the nitrates of calcium and strontium were evaporated. To the solution of strontium nitrate in a small beaker a few drops of sulphuric acid and then its volume of alcohol are added, whereby the strontium is precipitated as sulphate, in which form after twelve hours it is weighed and then tested spectroscopically as to freedom from calcium and barium.

### b. BY AMYL ALCOHOL.

Because of the slight solubility of strontium nitrate in amyl alcohol the method of Browning<sup>a</sup> does not appear to be adapted to the separation from calcium of the small amounts of strontium met with in rocks, though with barium the case is different, since its nitrate according to Browning is insoluble in absolute amyl alcohol.

## C. BEHAVIOR OF BARIUM.

Barium, after two ammonium-oxalate precipitations, will never be found with the ignited calcium and strontium in more than spectroscopic traces, unless originally present in excess of 3 or 4 milligrams, and very often only when in considerable excess.<sup>b</sup> If present with them, however, it will be separated with the strontium by ether-

<sup>a</sup> Am. Jour. Sci., 3d ser., vol. 43, 1892, pp. 50, 314.

<sup>b</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 16, 1894, p. 83; Chem. News, vol. 69, 1894, p. 147.

alcohol or amyl alcohol, and these two must then be treated by the ammonium-chromate method, given below, in order to arrive at the strontium. The barium is best estimated in a separate portion. (See "Barium" 13, p. 116.)

#### D. SEPARATION OF BARIUM FROM STRONTIUM.

Profiting by the experience of R. Fresenius and others, A. Skrabal and L. Neustadt<sup>a</sup> have shown in what manner alone a very nearly correct separation of barium can be made from calcium and strontium. The following solutions are requisite:

Ammonium bichromate, 100 grams to the liter.

Ammonium acetate, *a*, 300 grams, neutralized by ammonia, to the liter.

Ammonium acetate, *b*, 20 cm.<sup>3</sup> of *a* diluted to 1 liter.

The reaction of the acetate solutions should be alkaline rather than acid.

The method for the amounts used by the authors (0.135 gram BaO as the chloride and the same of strontium) is as follows:

To the neutral or weakly acid solution is added ammonium acetate (10 cm.<sup>3</sup> of solution *a*) in excess. The liquid is brought to boiling and while swirled about 5 cm.<sup>3</sup> of the bichromate solution is added. After settling and cooling the clear liquid is decanted through a filter and the precipitate washed by decantation with ammonium acetate (solution *b*) till the filtrate is no longer perceptibly colored (100 cm.<sup>3</sup> of wash solution). The beaker is placed under the funnel, the precipitate on the paper dissolved by warm dilute nitric acid, and the paper washed. More acid is then added to dissolve the rest of the precipitate, followed by ammonia till the precipitate forming again no longer redissolves. Ammonium acetate (10 cm.<sup>3</sup> of solution *a*) is now poured in, the liquid brought to boiling while kept in swirling motion, allowed to cool slowly, and the precipitate is washed by decantation with solution *b* of the ammonium acetate. The barium chromate is dried and weighed as such after ignition, the filter being burned separately.

The strontium may be thrown down from the combined filtrates by ammonia and ammonium carbonate, after concentration in presence of a little nitric acid, and weighed as carbonate; or the carbonate may be redissolved, precipitated by sulphuric acid and alcohol, and weighed as sulphate.

It is probable that for the small amounts encountered in rocks a single precipitation of the barium by bichromate will suffice, but this is not so when any considerable quantities are to be separated.

<sup>a</sup>Zeitschr. anal. Chemie, vol. 44, 1906, p. 742.

## E. OTHER METHODS OF DETERMINING THE WEIGHT OF CALCIUM.

## GENERAL STATEMENT.

Additional methods more or less in vogue for determining the calcium after precipitation as oxalate are the volumetric method by potassium permanganate and the conversion of the oxalate into carbonate, sulphate, or fluoride. None of these is applicable in rock analysis if it is desired to determine the strontium, except the second, but any one of them will apparently afford good results if no account is to be taken of that element. The methods have, however, not been tested in this laboratory, though the several gravimetric methods have been compared by O. Brunck,<sup>a</sup> who finds that duplicates by any one of them show better agreement than by the oxide method. Brief descriptions of them are therefore given below, as well as of the volumetric method.

It is to be said, however, regarding Brunck's determination of calcium as the oxide that no such variations as those reported by him are ever obtained in the Survey laboratory. Our duplicates by that method agree as well as his by the other methods. His high and varying results by the oxide method are probably to be attributed to the employment of a vertical instead of an inclined flame. The complete conversion of the oxide to sulphate by treatment with either sulphuric or hydrofluoric acid is, in my opinion, based on experience with the conversion of lead chloride to sulphate, by no means easily brought about. There will always be a tendency, where an insoluble compound is at once produced by action of one of the acids in question, for some of the original compound to be protected from conversion to the salt, even after two or more evaporations with fresh portions of acid.

It is further doubtful if the loss in weight of the crucible during blast ignition of the lime is really as great as he thinks. It is well known that barium oxide attacks platinum strongly when heated in contact with it, and it is certain that lime does so to a less but appreciable extent. That it does can readily be demonstrated by dissolving in hydrochloric acid lime that has been blasted in platinum for half an hour and precipitating and weighing the dissolved platinum. The amount recovered will nearly and sometimes quite equal the loss in weight of the crucible itself.

## a. VOLUMETRIC METHOD.

The washed oxalate is rinsed into a beaker and the filter is extracted with hot dilute sulphuric acid, the filtrate being caught in the beaker. It is not permissible to omit this extraction, for simple rinsing with water will not extract all of the oxalate held in the pores of the paper.

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<sup>a</sup> Zeitschr. anal. Chemle, vol. 45, 1906, p. 77.

ore acid is added if necessary, and the contents of the beaker are gested at a moderate heat till decomposition can be considered complete. The oxalic acid is then titrated warm at about 70° with permanganate, which has been standardized best against pure Iceland spar dissolved in hydrochloric acid and precipitated as the oxalate.

b. BY WEIGHING AS THE CARBONATE.

The oxalate is gently ignited to convert it to carbonate and is then gested with a few drops of a solution of ammonium carbonate. The liquid is evaporated cautiously and the residue gently ignited to just visible dull redness and weighed. The treatment with ammonium carbonate, etc., is repeated till constant weight is reached.

c. BY WEIGHING AS THE SULPHATE.

The strongly ignited oxalate is cautiously slacked with water and slight excess of sulphuric acid is added. The liquid is concentrated by evaporation and the excess of acid removed in the radiator (fig. 2, p. 29). The dry sulphate is then moderately ignited (to dull redness for a short time) and weighed, the treatment with acid, etc., being repeated to constant weight.

d. BY WEIGHING AS THE FLUORIDE.

The strongly ignited oxide is slacked with water and covered with hydrofluoric acid, the excess of which is removed on the bath and the residue ignited for a few moments and weighed. The treatment with acid, etc., is to be repeated till the weight becomes constant. According to Brunck this last method is simpler than the carbonate or sulphate method, and in the hands of the inexperienced leads to better results, though he regards the sulphate method as the best in experienced hands.

11. MAGNESIUM.

A. PRECIPITATION.

a. CONDITIONS NECESSARY FOR THE PRODUCTION OF A NORMAL PRECIPITATE.

There are few analytical procedures regarding which so much uncertainty has existed and still exists as that by which magnesium is precipitated as the ammonio-phosphate, notwithstanding the enormous mass of detailed work that has been done on the subject. The work of H. Neubauer,<sup>a</sup> confirmed in part and supplemented by F. A. Gooch and Martha Austin,<sup>b</sup> seemed to clear the ground fairly well. Neu-

<sup>a</sup>Zeitschr. angew. Chemie, 1896, p. 435.

<sup>b</sup>Am. Jour. Sci., 4th ser., vol. 7, 1899, p. 187; Chem. News, vol. 79, 1899, pp. 233, 244, and 255; Zeitschr. org. Chemie, vol. 20, 1899, p. 121.

bauer found that precipitation is complete even in the presence of large quantities of salts of ammonium, including the oxalate, but that the composition of the precipitate is largely affected by ammonium salts and also by the way in which the precipitation is made. Gooch and Austin show that the large amount of ammonia of 0.96 specific gravity (one-third the original volume of the solution) usually prescribed is not only unnecessary, but, in fact, disadvantageous, at least, until the precipitate has fully formed. The authors named agree that it is only by working under certain conditions—absence of any large excess of precipitant, of ammoniacal salts, and of ammonia—that a precipitate of normal composition is obtainable (see below). From their labors it would seem clear that the common way of adding the phosphate precipitant to the ammoniacal solution of the magnesium salt is not calculated to produce a precipitate of normal composition. The precipitant should be added to the acid solution of the magnesium, and ammonia should then be added in slight excess.

The precipitate as ordinarily formed differs from the normal in containing relatively more ammonium and less magnesium—for instance, an admixture of such a molecule as  $\text{Mg}(\text{NH}_4)_4(\text{PO}_4)_2$ —the result being that when ignited in the ordinary way too much magnesium is found because of formation of some metaphosphate. To obviate this error, Neubauer considers it absolutely necessary to blast the precipitate for half an hour and then to repeat the blasting for a second half hour to see if constant weight has been reached. The phosphate is then entirely pyrophosphate, which is quite unaffected by further blasting (see, however, B. a, p. 108). The intense heat has caused a decomposition of the metaphosphate with volatilization of  $\text{P}_2\text{O}_5$ , as follows:  $2\text{Mg}(\text{PO}_3)_2 = \text{Mg}_2\text{P}_2\text{O}_7 + \text{P}_2\text{O}_5$ . Neubauer worked with the usual excess of ammonia added after the bulk of the precipitate had formed, and it remains to be seen whether by precipitating and working according to Gooch and Austin the composition of the precipitate is always close enough to the ideal  $\text{MgNH}_4\text{PO}_4$  to obviate the necessity for blasting.

Gooch and Austin call attention to a modification proposed long since by Wolcott Gibbs,<sup>a</sup> whereby the phosphorus and magnesium salts are first boiled together in neutral solution for a few minutes and to the cooled solution ammonia is added. The results are said to be remarkably exact, but K. K. Järvinen<sup>b</sup> reports to the contrary. This chemist obtained a normal precipitate by either of two methods of his own, of which he regards the following as more convenient and reliable, because ammonium salts exert no influence on the result. It is likewise applicable to the precipitation of phosphoric acid by magnesium mixture.

<sup>a</sup> Am. Jour. Sci., 3d ser., vol. 5, 1873, p. 114.

<sup>b</sup> Zeitschr. anal. Chemie, vol. 44, 1905, p. 335.

The hot magnesium solution, containing at least 2 to 3 grams of ammonium salts (to prevent precipitation of magnesium hydroxide) and 20 to 30 cm.<sup>3</sup> of 10 per cent ammonia solution for 0.4 gram  $\text{Mg}_2\text{P}_2\text{O}_7$ , is heated to boiling and the precipitant is added slowly. Then more ammonia is added and the solution allowed to cool. A coarsely crystalline precipitate forms which, it is claimed, consists entirely of dimagnesium-ammonium phosphate. This procedure of Arvinen was employed where but a single precipitation of the magnesium was intended, and it does not seem to be adapted for a second precipitation. As this is always called for in rock analysis, it does not appear best to adopt his procedure for our purposes.

#### b. PRECIPITATION.

*α. First precipitation.*—This is made without special precautions in the filtrate from the first calcium-oxalate separation (p. 101) by sodium-ammonium-hydrogen phosphate (microcosmic salt)<sup>a</sup> in indefinite decided excess and without the great excess of ammonia usually prescribed. It is not necessary to first remove ammoniacal salts unless very little magnesium is present, and then only in order to hasten precipitation. The facts that large quantities of these salts retard precipitation and that the composition of the precipitate is affected by them as well as by the way in which the precipitation is made are of importance only when a single precipitation is to be made or in the case of two or more.

Continued agitation of the liquid by a mechanical stirrer is of advantage in greatly shortening the time of complete precipitation, which is in general shorter the greater the quantity of magnesium present.

*β. Second precipitation.*—Platinum sulphide usually strongly contaminates the separated phosphate, but this matters not, as it remains on the filter when the phosphate is redissolved in hydrochloric acid, of which not more than the amount really needed should be used. The solution thus obtained is united with that of the residue from evaporation and ignition of the second filtrate from the calcium oxalate (10. A. a, p. 101), and is diluted if necessary. A few drops of sodium-ammonium-phosphate solution are now added, and ammonia, drop by drop, in slight excess, with constant stirring till the crystalline precipitate has well formed. Once formed a few cubic centimeters more of ammonia are to be added.

<sup>a</sup> The objection that has been made by one writer to the use of this salt instead of disodium-hydrogen phosphate is, so far as our experience teaches, entirely groundless.



## B. METHODS OF COLLECTING AND IGNITING THE PRECIPITATE.

## a. ORDINARY PROCEDURES.

Were it not for the necessity of examining the precipitate for impurities after ignition (C., p. 109), it were best collected in a Gooch crucible, provided the asbestos felt is well constructed and not of the serpentine variety so largely on the market. It is therefore well to use an ordinary crucible of platinum. Neubauer ignites slowly in platinum after drying, without removing from the paper, and applies the blast only when the carbon has been wholly burned off.<sup>a</sup> The danger of attack of the platinum by thus igniting the precipitate in contact with the paper is extremely slight, though it has happened once or twice in our experience that a crucible has been ruined by so doing. Why reduction should have occurred in these cases and not in the hundreds of others is not known.

The precipitate, wrapped in its moist paper, is best dried in the crucible itself, the paper then slowly charred without allowing it to ignite, the carbon burned off over a gradually increasing flame, and finally a weak blast applied for a long time and repeated to constant weight to insure volatilization of any excess of  $P_2O_5$  over and above that required for the pyrophosphate formula (see A. a, p. 105).

The experience of the Survey chemists was for a time decidedly adverse to the blasting of magnesium pyrophosphate as prescribed by Neubauer, whether obtained by precipitation of magnesium or phosphate solutions. If the weights were not large the salt fused in part or wholly, and when this occurred reduction seemed to be rapid and the crucible was rather strongly attacked, as shown by its loss in weight after dissolving out the pyrophosphate. Presumably this effect was caused by a blast of higher temperature than that employed by Neubauer, who is very emphatic in his assertion that neither pyrophosphate nor crucible suffers the slightest loss when blasted for hours. This seems to be true with a blast of but moderate temperature, giving less than  $1,100^\circ$  in the crucible. What effect the presence of slight amounts of foreign phosphates may have in increasing the fusibility of magnesium pyrophosphate is unknown. Järvinen found that his precipitates obtained by the Gibbs method fused readily and gave too high results, and further that  $P_2O_5$  did seem to volatilize slowly from  $Mg_2P_2O_7$  over the blast.

## b. MODIFICATION OF ULBRICHT.

At one time the procedure first recommended by R. Ulbricht, later by K. Broeckmann, and also by L. L. de Koninck, was used. It consists in dissolving the ammonium-magnesium phosphate off the filter

<sup>a</sup> Zeitschr. anal. Chemie, vol. 33, 1894, p. 362.

with nitric acid, collecting the filtrate in a weighed crucible, evaporating the contents to dryness,<sup>a</sup> and subsequently igniting, the product being presumably pyrophosphate. But it was soon observed that the ignited salt, especially when large in amount, does not always dissolve completely in hydrochloric acid, but that sometimes a white residue is left in light lumps which appears to be quite insoluble in acids. This residue contains no silica, but only the constituents of a magnesian phosphate, and it may be a peculiar metaphosphate. Whether its appearance is due to an abnormal composition of the original magnesian precipitate or to conceivable change during evaporation in the crucible with nitric acid remains to be determined. Until this is done the employment of this method of igniting is not to be recommended.

### C. CONTAMINATION BY AND REMOVAL OF BARIUM, CALCIUM, AND MANGANESE.

#### a. BARIUM.

Barium phosphate will not contaminate the second magnesian precipitate unless there are notable amounts of barium in the rock, in which case it must be removed by sulphuric acid prior to the final precipitation of the magnesium.

#### b. CALCIUM.

Calcium, however, is probably never absent, and has to be estimated and allowed for as follows:

To the ignited pyrophosphate, dissolved in but slight excess of hydrochloric acid, is added ammonia to alkalinity, and then acetic acid, drop by drop, till the solution, which should measure 10 to 30 cm.<sup>a</sup> and not be hot, clears. It now and then happens that a little flocculent matter fails to dissolve. This is to be removed, ignited, and subtracted from the original weight, if proved to be free from magnesium, which it often contains (see c, p. 110). It is likely to consist, in great part or wholly, of phosphates of iron or manganese, or both, and shows often a reddish color on ignition. If an excess of acetic acid has been used, this is cautiously removed by ammonia. Then a drop or two of solution of ammonium oxalate is added, and the small beaker is set aside for twelve hours if necessary. Almost invariably a small precipitate soon shows itself, which, if fine grained and nonadherent to the glass, may be regarded as pure calcium oxalate; otherwise it contains, or may largely consist of, magnesium oxalate. It is in that case to be collected, ignited, redissolved, and reprecipitated. Its final weight, averaging perhaps one-half milligram, is to be added to that of the lime already found and subtracted as tricalcium phosphate (not pyrophosphate) from that of the magnesium pyrophosphate, in order

<sup>a</sup> A pink color of varying intensity almost invariably becomes apparent as the mass approaches dryness, a most delicate test for the traces of manganese which always escape precipitation by ammonium sulphide or bromine.

to arrive at the true figure for magnesia. This separation, to be satisfactory, requires great care.

#### C. MANGANESE.

A separation from one another of the iron, aluminum, and manganese which the small flocculent precipitate mentioned in b may contain is not worth the trouble, careful prior treatment being presupposed. The first two constituents may have been introduced since the original precipitation of the iron and aluminum, and their amount in terms of those elements is very small. But the amount of manganese it contains may be a sensible proportion of the whole of that element. The precipitate should therefore be brought into nitric-acid solution. The solution containing the magnesium is also manganiferous. It is to be evaporated to dryness, the volatile salts expelled by ignition, and the residue again evaporated two or three times with a few drops of nitric acid to certainly remove all traces of chlorine, and united with the small solution above mentioned. The manganese is now determined colorimetrically, as in 9.C.b, page 99, and its amount added to that already found, unless the total has been ascertained from a separate portion of the sample.

The distribution of the manganese over the alumina, lime, and magnesia, when ammonia has been employed for the precipitation of the alumina and no attempt has been made to remove the manganese at all, is shown in the table on page 96.

#### D. LIABILITY TO ERROR IN THE AVERAGE MAGNESIUM DETERMINATION.

Experience has shown that the majority of analysts are pretty sure to make a very serious plus error in determining moderate amounts of magnesia, such as are found in limestones and Portland cements. The causes of this error will be sufficiently apparent after a careful perusal of the foregoing pages. Everyone ought to make the tests for foreign contamination just described, if only to convince himself of the reality of the error even after very careful work.

### 12. TITANIUM.

#### A. COLORIMETRIC DETERMINATION WITH HYDROGEN PEROXIDE.

##### a. DESCRIPTION OF THE METHOD (WELLER'S).<sup>a</sup>

The method consists in comparing the color of a known bulk of solution to be tested with that of a standard solution of titanium sulphate, both having been fully oxidized by hydrogen peroxide. The strength of the peroxide should be approximately measured by titration with permanganate on opening a fresh bottle and again after a few weeks, otherwise very serious error may arise through its deterioration.

<sup>a</sup> Ber. Deutsch. chem. Gesell., vol. 15, 1882, p. 2593.

Mere traces of hydrofluoric acid, in either the peroxide or the titanium solution, render this method inexact,<sup>a</sup> hence care should be exercised as to the character of the peroxide, which, as sold in the market, often contains fluorine. For method of testing see page 36.

Dunnington<sup>b</sup> has pointed out the necessity for the presence of at least 5 per cent of sulphuric acid in solutions which are to be thus tested for titanium, in order, as he concludes, to prevent partial reversion to metatitanic acid, which does not give a color with hydrogen peroxide. The standard solution of titanium sulphate,<sup>c</sup> holding conveniently about 1 centigram  $\text{TiO}_2$  in 10 cm.<sup>3</sup>, equivalent to 1 per cent of  $\text{TiO}_2$  in 1 gram of rock, contains, therefore, 5 per cent or more of sulphuric acid. Of this, 10 cm.<sup>3</sup> are mixed with a sufficiency of hydrogen peroxide (2 cm.<sup>3</sup> of most commercial brands is ample) and diluted to 100 cm.<sup>3</sup> in a measuring flask.

Titanium can be determined, as a rule, most conveniently in the solution which has served for the titration of total iron ( $\gamma$ , p. 92). This, having been evaporated, if necessary, to less than 100 cm.<sup>3</sup>, is to be fully oxidized with hydrogen peroxide, and if the color is less intense than that of the standard, is made up to 100 cm.<sup>3</sup> with dilute sulphuric acid in a measuring flask, and mixed; otherwise, in a flask of sufficient size to insure that its color shall be less intense. One of the rectangular glasses (fig. 5, p. 32) being filled with the solution to be tested, 10 cm.<sup>3</sup> of the diluted standard are run into the other from a burette, and water is added from a second burette until there is no distinction as to color. A second and a third portion of the standard can be run in and diluted and the mean of several determinations struck, when a simple calculation gives the percentage of  $\text{TiO}_2$  in the rock, the amounts in the two solutions being directly as their volumes.

If the convenient but expensive Soleil-Duboscq colorimeter is used, or the modification of O. Schreiner (fig. 6, p. 33), or the simple Nessler tubes, it is of course unnecessary to dilute the rock solution to the extent above required, should it be stronger than the standard. Experience has shown, however, that differences can not be sharply estimated in strongly colored solutions, and that the results are much more satisfactory when the color intensity is not much if any greater than that given by a standard of the above concentration. For

<sup>a</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 17, 1895, p. 718; Chem. News, vol. 72, 1895, p. 158; Bull. U. S. Geol. Survey No. 167, 1900, p. 56.

<sup>b</sup> Jour. Am. Chem. Soc., vol. 13, 1891, p. 210.

<sup>c</sup> Potassium-titanium fluoride,  $\text{K}_2\text{TiF}_6$ , best serves as the starting point for its preparation. A quantity of this is recrystallized from boiling water once or more times, dried, and preserved in a glass-stoppered bottle. Enough of it to make one-half to 1 liter of the standard sulphate solution is weighed into a platinum dish and evaporated several times, without bringing to dryness, with strong sulphuric acid until the fluorine is completely expelled. The residue is then taken up with water containing enough sulphuric acid to make 5 per cent of the latter when fully diluted. Two 50-100 cm.<sup>3</sup> portions of the prepared solution are then further diluted, boiled, and precipitated with ammonia. The precipitates are collected on paper, washed with hot water till free from alkali, ignited moist in the filter, blasted, and weighed. Duplicates should agree almost exactly.

the percentages of titanium found in rocks, clays, and soils, usually under 1 per cent, but rising to 2 or even 3 per cent or more occasionally, the colorimeter method gives results which are fully equal to those of the best gravimetric method, besides being a great time saver.

#### b. CORRECTION FOR THE COLOR OF FERRIC SULPHATE.

The error introduced by iron, in consequence of the yellowish color of its sulphate solution, is practically negligible unless its percentage is high; then either the iron must be removed, as described under B.a.α, page 113, prior to making the color test, or correction should be applied for known amounts of ferric sulphate in solutions of the requisite dilution.

The exact correction to be applied in such cases is difficult of determination because of the impossibility of matching the colors of titanium-peroxide solutions with those of ferric sulphate; but tests made go to show that the coloring effect of 0.1 gram of  $\text{Fe}_2\text{O}_3$  in 100 cm.<sup>3</sup> of 5 per cent sulphuric-acid solution is about equal to 0.2 milligram of  $\text{TiO}_2$  in 100 cm.<sup>3</sup> when oxidized by hydrogen peroxide. This amounts to a correction of only 0.02 per cent on one gram of rock containing the unusual amount of 10 per cent  $\text{Fe}_2\text{O}_3$ .

W. A. Noyes<sup>a</sup> overcomes the difficulty mentioned in the last paragraph by adding to the standard solution approximately as much ferric iron as will give it, after the colors are matched, the same iron strength as the test solution. This could not be done with the apparatus depicted in fig. 6 (p. 33), though practicable with Nessler tubes. In using the Survey colorimeter (fig. 5, p. 32), it would perhaps be best to make first an approximate determination of the titanium in order to find the final volume of the standard after dilution, then to make an exact determination, having added to a fresh portion of the standard the requisite amount of iron salt, preferably in the form of ferric-ammonium alum. The amount to be used is calculated as follows: Let  $A$  be the number of cubic centimeters of the test solution,  $a$  of the standard used,  $b$  of the water needed to dilute the standard in the preliminary test,  $p$  the weight of iron in terms of  $\text{Fe}_2\text{O}_3$  in  $A$ , and  $x$  the weight of iron in terms of  $\text{Fe}_2\text{O}_3$  to be added to each cubic centimeter of water used for diluting the standard in the final test.

Then  $x = \frac{a+b}{Ab} p$ . For all practical purposes it would probably be sufficient to add the requisite amount of iron salt in solid form to the standard after having diluted it nearly enough to match the test solution, and after it has dissolved to proceed with the test. But this procedure has the disadvantage that each comparison would necessitate the weighing out of a separate portion of iron salt, whereas by the former method the number possible would be limited only by the

<sup>a</sup> Jour. Anal. and Appl. Chem., vol. 5, 1891, p. 39.

stock of standard and of the prepared ferric-ammonium sulphate solution.

#### C. ALTERNATIVE MODE OF PREPARING THE TEST SOLUTION.

As said above (pp. 92,  $\gamma$ , and 110, 12. A. a), the solution that has been used for volumetric determination of total iron can most conveniently be used for the colorimetric determination of titanium, but if desired this can, of course, be made on some other portion of rock powder. At one time it was the practice in this laboratory to combine it with the determination of barium, as described in Bulletin No. 148, by decomposing the powder by sulphuric and hydrofluoric acids,<sup>a</sup> expelling the latter by repeated evaporations with sulphuric acid, taking up with dilute sulphuric acid,<sup>b</sup> filtering from barium sulphate, etc., and estimating the titanium colorimetrically in the filtrate. The expulsion of fluorine must be thorough, or else the titanium result will be low, as already stated (p. 111), and it is not always easy to effect this complete removal, though the time required to do so seems to be in no slight degree dependent on the nature of the fluorides to be decomposed. Long after every trace of fluorine seems to be gone, the formation of a crust on the evaporating solution sometimes allows an accumulation of enough hydrofluoric-acid gas to become plainly manifest to the smell on breaking the crust.

#### B. GRAVIMETRIC METHODS.

##### a. GOOCH'S METHOD.

*a. Description of the method.*—When titanium is present in excess of 4 to 5 per cent and whenever for any reason it is desired to employ a gravimetric method, among the few that have been thoroughly tested that of Doctor Gooch<sup>c</sup> is unequaled. With one or two minor modifications introduced by Dr. T. M. Chatard,<sup>d</sup> it is as follows:

Any solution of the rock freed from silica can be used, and the first step is to remove the iron. This is best done, after adding tartaric acid and reducing the iron by means of hydrogen sulphide to the ferrous condition, by rendering the solution ammoniacal and introducing more hydrogen sulphide. If the iron is not thus reduced before precipitation, titanium will be in part thrown down also.<sup>e</sup> The amount

<sup>a</sup> It is to be borne in mind that evaporation with hydrofluoric acid alone results in loss of titanium by volatilization, but that there is no loss if excess of sulphuric acid is also present.

<sup>b</sup> With acid rocks solution is very complete, and it can be made nearly so with the most basic by transference to a small beaker and gentle boiling. The residue thus obtained may contain, besides barium sulphate, a little calcium sulphate, zircon, andalusite, topaz, and possibly a trace of titanium in some form. It is therefore to be thoroughly fused with sodium carbonate, leached with water, fused with potassium pyrosulphate, dissolved in dilute sulphuric acid, filtered, and the filtrate added to the main one. The insoluble matter will now be chiefly barium sulphate, for the further treatment of which see 13. A (p. 116).

<sup>c</sup> Proc. Am. Acad. Arts Sci., n. s., vol. 12, p. 435; Bull. U. S. Geol. Survey No. 27, 1886, p. 16; Chem. News, vol. 52, 1885, pp. 55, 68.

<sup>d</sup> Am. Chem. Jour., vol. 13, 1891, p. 106; Bull. U. S. Geol. Survey No. 78, 1891, p. 87; Chem. News, vol. 63, 1891, p. 267.

<sup>e</sup> Cathrein, A., Zeitschr. f. Kryst., vol. 6, 1882, p. 243; vol. 7, 1883, p. 250.

of tartaric acid is to be gaged according to the combined weights of the oxides to be held by it in solution, and three times this weight is ample. After removing the iron sulphide by filtration—little washing suffices, because of the relatively small amount of titanium commonly present—the tartaric acid is destroyed as follows:

Potassium permanganate to the extent of two and one-half times the weight of the tartaric acid used is made into a strong solution, and to the ammoniacal filtrate from the iron sulphide enough sulphuric acid is introduced to leave some excess after all the permanganate has been reduced. After expulsion of hydrogen sulphide by boiling, the permanganate is added gradually to the hot solution contained in a large beaker or flask. A vigorous reaction ensues. When a permanent brown precipitate of manganic hydrate appears, the tartaric acid has been fully broken up, and the precipitated manganese is to be redissolved by a few drops of ammonium bisulphite or of sulphurous acid solution.

Ammonia is then added in slight excess, followed at once by acetic acid in considerable excess, and the boiling is continued for a few minutes. Thereby the titanium is freed from most of the alumina, and from lime and magnesia if they had not been earlier removed, also from most of the manganese introduced. The precipitate is filtered and washed with water containing acetic and sulphurous acids, then ignited, fused thoroughly with sodium carbonate, and leached with water to remove phosphoric acid and most of the remaining alumina. The residue is again ignited and fused with sodium carbonate. To the cooled melt in the crucible strong sulphuric acid is to be added, wherein it dissolves readily by aid of gentle heat. This solution is to be poured into a small volume of cold water and the platinum it contains precipitated by hydrogen sulphide at or near boiling temperature. After filtering and cooling, ammonia is added till the titanium is just precipitated, and a measured volume, containing a known weight of absolute sulphuric acid, is then added—just enough to redissolve the precipitate. The solution is then made up with acetic acid in such amount that the final bulk shall contain from 7 to 11 per cent of absolute acid, and then enough solid sodium acetate is stirred in to more than take up the sulphuric acid introduced. On rapidly bringing the liquid to ebullition the titanium is precipitated in flocculent and easily filterable condition, and the precipitation is complete after a minute's boiling, provided all the prescribed conditions have been followed and zirconium is absent.

The precipitate is washed first with acetic acid of 7 per cent strength and then with hot water. After fifteen to twenty minutes' ignition over a good burner it is in condition for weighing and will lose no more weight over the blast lamp. For large amounts of titanium a repetition of the sodium-carbonate fusion, etc., should be made. The

tual carrying out of all these operations, when once the method is understood, requires much less time than the detailed description would indicate.

*β. Gooch's method not directly applicable to rocks containing zirconium.*—Prior to the adoption of the colorimetric method, Doctor Gooch's was invariably used in this laboratory. Occasional inability to secure clean and complete precipitation by it was experienced, especially with a certain series of rocks rather poor in titanium. Long search showed the difficulty to be due to the presence of zirconium, which acts as a marked preventive of the precipitation of titanium by boiling in an acetic-acid solution under the conditions of the Gooch method.

The above rocks were found to contain up to 0.2 per cent of  $ZrO_2$ , and this amount was able to prevent precipitation of 0.3 per cent of  $O_2$ . The titanium which came down in excess of this amount did not settle out in flocculent condition, as happens when zirconium is not present, and it was difficult to filter. After the removal of the zirconium, however, in the manner to be hereafter described (14. A, 118), no difficulty was experienced in precipitating all the titanium with the usual ease.

#### b. BASKERVILLE'S METHOD.

C. Baskerville<sup>a</sup> has proposed the separation of titanium from iron and aluminum by boiling the neutralized solution of the chlorides for a few minutes in presence of sulphurous acid. The test separations as given by him are sharp, and a single precipitation is said to suffice, the titanium being free from iron and easily filterable. This last statement and the ready precipitability are fully confirmed by the experiments of the writer on titaniferous iron ores, but, although the titanium is completely thrown out, it carries with it a little iron, for instance, about 0.25 per cent  $Fe_2O_3$  with 8 to 10 per cent  $TiO_2$ . Zirconium would probably be likewise precipitated (14. B, p. 120) and phosphorus perhaps also, but this last point has not been investigated, whether has the applicability of the method to aluminous rocks been tested.

#### c. METHOD OF DITTRICH AND POHL.

Attention has been directed (8. C. b, p. 94) to the error resulting from attempting to separate aluminum from titanium by either fused or dissolved sodium hydroxide in absence of appreciable amounts of iron. When iron is present, the procedure given by Dittrich<sup>b</sup> may be used. It involves fusion in a silver crucible of the ignited oxides of aluminum, iron, etc., obtained in the usual way, with sodium

<sup>a</sup>Jour. Am. Chem. Soc., vol. 16, 1894, p. 427.

<sup>b</sup>Anleitung zur Gesteinsanalyse, 1905, pp. 9-31; also Dittrich, M., and Pohl, R., Zeitschr. anorg. Chemie, L. 43, 1905, p. 236.



hydroxide, re-solution of the insoluble matter in hydrochloric acid, precipitation of the iron as sulphide from an ammoniacal tartrate solution, destruction of the tartrate, precipitation of titanium (and zirconium) by ammonia, and determination of the titanium colorimetrically after ascertaining the combined weight of the two oxides.

C. SUPERIORITY OF THE COLORIMETRIC AND GOOCH METHODS OVER THE OLDER ONES.

In view of the good results obtainable by the colorimeter method in all cases, and by the Gooch method in the absence of zirconium, it is inexplicable that the old method of precipitation by many hours' boiling in a nearly neutral sulphate solution in presence of sulphurous acid should still find adherents in any part of the world.

13. BARIUM (ZIRCONIUM, RARE EARTHS, TOTAL SULPHUR, CHROMIUM).

These five constituents can with great economy of time and labor be determined in the same portion of rock powder, of which a 2-gram portion should be used, though if vanadium is to be looked for it will be best to determine it and chromium in a different portion (18.C, p. 127). It has been said above (10. C, p. 102) that only in very exceptional cases will barium be found with the calcium and strontium after two or possibly three precipitations of the latter as oxalate, since it passes into the filtrates with the magnesium, whence it may be obtained as sulphate after removal of ammoniacal salts. Addition of some alcohol insures also the recovery of traces of strontium if the rocks are very rich in it; but it is unsafe to regard the amount of barium thus separated from the magnesium as representing the total amount in the rock. It will almost always be found lower than the truth, probably for the reason that there are opportunities during the analysis for slight losses.

A. THE GENERAL METHOD.

Decomposition is effected by fusing the 2-gram portion with sulphur-free sodium carbonate and an amount of niter insufficient to injure the crucible first over the Bunsen flame, then over the inclined blast, the crucible, being fitted snugly into a hole in a disk of platinum foil or asbestos board (fig. 3, p. 30) to prevent access of sulphur from the gas flame. In case sulphur is not to be regarded, the niter and disk are omitted. After thorough disintegration of the melt in water, to which a drop or two of methyl or ethyl alcohol has been added for the purpose of reducing manganate, the solution is filtered and the residue washed with a very dilute solution of sodium carbonate free from bicarbonate. This is to prevent turbid washings. A yellow color in the filtrate indicates chromium.

(For the further treatment of the filtrate see "Sulphur," 24.B, p. 160, and "Chromium," 17.B, p. 124.)

The residue is washed from the paper into a small beaker without removing the filter from the funnel and digested with little more than enough warm dilute sulphuric acid to effect solution of all soluble constituents (stronger acid may be used and in larger amount if barium only is sought). A few drops of sulphurous acid are added at the same time to effect solution of the brown hydroxide of manganese. There will remain undissolved more or less residue. Care must be taken not to digest so long with acid as to cause gelatinization of the dissolved silica. The liquid is passed through the original filter and collected in an Erlenmeyer flask of 100 to 150 cm<sup>3</sup>. The paper with its contents after washing is ignited, evaporated with hydrofluoric and sulphuric acids together, and the final residue taken up with a little hot dilute sulphuric acid. All the barium will remain undissolved, besides some of the strontium and perhaps a good deal of calcium. This is collected on a small filter and the filtrate added to the former one, which now contains all the zirconium and rare earths. (For its further treatment see 14.A, p. 118, and 15.A, p. 120.)

The ignited residue last obtained is fused with sodium carbonate, leached with water, and the residue dissolved off the filter by a few drops of hydrochloric acid, from which solution the barium is thrown out by a large excess of sulphuric acid. A single solution of the ignited barium sulphate in concentrated sulphuric acid and reprecipitation by water suffices to remove traces of calcium which might contaminate it if the rock was one rich in calcium, and even strontium is seldom retained by it in quantity sufficient to give concern. Should this be the case, however, which will occur when the SrO and BaO are together in the rock in, roughly speaking, 0.2 and 0.4 per cent respectively, the only satisfactory way is to convert the sulphates into chlorides and to apply to the mixture the ammonium-chromate method of separation (10.D, p. 103).

Barium and strontium sulphates can be brought into a condition for testing spectroscopically by reducing for a very few moments the whole or part of the precipitate on a platinum wire in the luminous tip of a Bunsen burner, then moistening with hydrochloric acid, and testing in the usual manner.

The procedure outlined in the foregoing paragraphs for the estimation of calcium, strontium, and barium in silicate rocks is the one which long experience has shown to be best adapted for securing the most satisfactory results with a minimum expenditure of time.<sup>a</sup> Even where no attempt is made to separate contaminating traces of strontium and barium one from the other, the error is usually of no

<sup>a</sup>For details consult Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 16, 1894, p. 83; Chem. News, vol. 69, 1894, p. 147.

great consequence, for an absolute error of even 25 per cent in a substance constituting only 0.1 or 0.2 per cent of a rock is ordinarily of small moment compared with the ability to certify to its presence with approximate correctness.

With such small amounts of barium as are usually found in rocks it is doubtful if Mar's<sup>a</sup> method for the separation of barium from calcium and magnesium by the solvent action of concentrated hydrochloric acid mixed with 10 per cent of ether on the chlorides could be conveniently applied here, although for larger amounts the method would seem to be accurate and easily executed. Moreover, it would probably not entirely remove contaminating strontium, and hence offers no advantage.

#### B. THE METHOD WITHOUT REGARD TO ZIRCONIUM AND SULPHUR.

If zirconium and sulphur are not to be looked for, the simplest procedure is to decompose the powder by sulphuric and hydrofluoric acids (see A.c, p. 113, under "Titanium"), and to complete the purification of the barium sulphate thus obtained in the manner described in the fourth paragraph of A above.

### 14. ZIRCONIUM.

This element is rarely looked for by chemists, though shown by the microscope to be one of the most constant rock constituents, usually in the form of zircon, in which occurrence its amount can be approximately judged and a chemical test rendered almost unnecessary; but sometimes it occurs in other minerals, and is then unrecognized under the microscope. It may in most exceptional cases be present up to a few per cent of the rock, rarely reaches 0.2 per cent, and is usually much less than 0.1 per cent.

#### A. AUTHOR'S METHOD.

For its detection and estimation in such cases, or whenever a search for it seems called for, the following procedure, based on a method used by G. H. Bailey,<sup>b</sup> has been devised, which serves, when carried out with care, to detect with certainty the merest trace—0.02 per cent, for instance—in 1 gram.

The preliminary treatment of the rock powder has been fully given under "Barium" (13.A, p. 116), where the separation from barium has been described and also the concentration of the zirconia in a small amount of very dilute sulphuric solution. This should probably not contain much above 1 per cent of sulphuric acid, though the actually permissible limit has not been established. To the solution, which should be in a small flask, is now added hydrogen peroxide to oxidize

<sup>a</sup> *Phil. Mag.*, vol. 43, 1892, p. 521.

<sup>b</sup> *Jour. Chem. Soc.*, vol. 49, 1886, pp. 149, 481.

the titanium, and then a few drops of a soluble orthophosphate solution. The flask is set aside in the cold for twenty-four to forty-eight hours. If the color bleaches after a time, more hydrogen peroxide should be added. Under these circumstances the zirconium is thrown out as phosphate and collects as a flocculent precipitate, which at this stage is not always pure. (For the treatment of the filtrate see "Rare-earth metals," 15.A, p. 120.) No matter how small or insignificant, it is collected on a filter, ignited, fused with sodium carbonate, leached with water, the filter again ignited, fused with very little acid potassium sulphate, brought into solution in hot water with a few drops of dilute sulphuric acid, poured into a flask or beaker of about 20 cm.<sup>3</sup> capacity, a few drops of hydrogen peroxide and of sodium phosphate are added, and the flask set aside. Titanium is now almost never present, and the zirconium soon appears as a colorless flocculent precipitate, which can be collected and weighed as phosphate. For the small amounts usually met with it is safe to assume that it contains 50 per cent of  $ZrO_2$  (51.8 by theory). If the amount is rather large, it may be used with sodium carbonate, leached, ignited, fused with acid potassium sulphate, reprecipitated by ammonia, and weighed as  $ZrO_2$ . Certainty as to its identity can be had by again bringing it into solution, reprecipitating by ammonia, dissolving in hydrochloric acid, evaporating to a drop or two, and testing with turmeric paper or by a microchemical reaction. With the very smallest amounts no color can be obtained by this turmeric-paper test, which, however, responds readily to as little as 1 milligram of dioxide and with proper care for as small an amount as 0.3 milligram (Dr. H. N. Stokes). No element other than thorium is ever likely to contaminate the zirconium thus reprecipitated.

In Bailey's experiments the precipitation was not made by addition of a phosphate, but is said to be due solely to the hydrogen peroxide, the precipitate being a hydrated peroxide,  $Zr_2O_5$ , or  $ZrO_3$ .<sup>a</sup> My own efforts as well as those of others to secure a precipitate in acid solutions of zirconium sulphate by hydrogen peroxide alone have been unsuccessful, except in very concentrated solutions and with 30-per cent peroxide. The ability to obtain the zirconium free from phosphoric acid would certainly be a great improvement on the method described above.

Were it not for the necessity of precipitating the zirconium in a weakly acid solution, its separation could be made in the same portion in which the titanium is colorimetrically determined.

#### B. OTHER METHODS.

G. Streit and B. Franz<sup>b</sup> claim to secure complete separation of titanium from iron and zirconium by boiling the neutralized solutions of the sulphates with a large excess (50 per cent) of acetic acid. The

<sup>a</sup>Chem. News, vol. 60, 1889, p. 6.

<sup>b</sup>Jour. f. prakt. Chemie, vol. 108, 1889, p. 65.

method has been from time to time recommended, but without any data showing its value. The single separation made by Streit and Franz was far from perfect.

J. T. Davis<sup>a</sup> separated zirconium sharply from aluminum, but not from iron, by precipitation as an oxyiodate in a boiling neutralized solution of chlorides, but the method is hardly applicable for rock analysis.

C. Baskerville<sup>b</sup> has proposed a method for the separation of zirconium from iron and aluminum similar to his method for the separation of titanium from those elements (12.B.b, p. 115). It is based on the precipitability of zirconium by boiling the neutralized chloride solution for two minutes in presence of sulphurous acid, and seems to be excellent. As titanium is always present and is presumably quantitatively thrown down also, the two would have to be separated by hydrogen peroxide. No tests as to the availability of the method for separating the small amounts met with in rock analysis have been made.

The method of M. Dittrich and R. Pohl for determining zirconium by difference, after ascertaining gravimetrically the combined weight of zirconium and titanium and by colorimetry that of titanium, has been outlined on page 115, under c.

#### 15. RARE-EARTH METALS OTHER THAN ZIRCONIUM.

For the cases in which it may be necessary to look for rare earths other than zirconia, the following procedures will prove satisfactory and not at all difficult.

##### A. USUAL METHOD.

The filtrate from the zirconium phosphate (14.A, p. 118) or, if zirconium is not to be determined, the earlier solution (13.A, p. 116), is treated with excess of potassium hydroxide to precipitate the rare earths, and ferric and titanous oxides, while retaining the silica and alumina in solution. After settling the liquid is decanted and the precipitate is washed once or twice by decantation and slightly on the filter. The precipitate is washed from the paper into a small platinum dish, treated with hydrofluoric acid, and the liquid evaporated nearly to dryness. A little water with a few drops of hydrofluoric acid is added and the insoluble rare-earth fluorides (crude) collected on a small filter held by a perforated platinum or rubber cone and washed with water acidified with the same acid. The precipitate is washed into a small platinum dish and evaporated to dryness with sulphuric acid, the paper being burned and added before expulsion of the acid. The sulphates are dissolved in dilute hydrochloric acid, the earth hydroxides precipitated by ammonia, redissolved in hydro-

<sup>a</sup> *Am. Chem. Jour.*, vol. 11, 1889, p. 27.

<sup>b</sup> *Jour. Am. Chem. Soc.*, vol. 16, 1894, p. 475; *Chem. News*, vol. 70, 1894, p. 57.

chloric acid, the solution evaporated to dryness, and the residue heated with a few drops of a solution of oxalic acid. Anything that may be present except the rare earths dissolves readily, these remaining as insoluble oxalates. If there is a doubt as to the complete separation of the alkaline earths the ignited oxalates may be dissolved in hydrochloric or nitric acid, precipitated by ammonia, redissolved in whichever one of the acids may have been used, the solution evaporated to dryness, the oxalates reprecipitated, ignited, and the resulting oxides weighed.

The amounts are too small to permit separation into further groups, but the presence of cerium can be readily shown if a strong yellow color develops on heating the ignited oxides with strong sulphuric acid.

#### B. ALTERNATIVE METHOD.

The rock powder is thoroughly decomposed by several partial evaporations with hydrofluoric acid, the transparent precipitate of silico-fluorides and of the fluorides of all earth metals except zirconium is collected on a platinum cone, washed with water acidulated by hydrofluoric acid, washed back into the dish or crucible, and evaporated with enough sulphuric acid to expel all fluorine. The filter is burned and added. By careful heating the excess of sulphuric acid is removed and the sulphates are taken up by dilute hydrochloric acid. The rare earths, with perhaps some alumina, are then precipitated by ammonia, washed, redissolved in hydrochloric acid, and evaporated to dryness, then taken up with water and a drop of hydrochloric acid, and only enough ammonium acetate to neutralize the latter added, followed by oxalic acid (not ammonium oxalate, which would fail to precipitate thorium). In this way as little as 0.03 per cent of rare earths has been found when working on not more than 2 grams of material.

### 16. PHOSPHORUS.

It is sometimes possible to extract all phosphorus from a rock by simple digestion with nitric acid, but quite as often, if not oftener, this fails; hence the necessity for resorting to one of the longer methods of extraction detailed below. Whatever method is used great care is required in order to secure accurate results.

#### A. PROCEDURE WHEN MATERIAL IS AMPLE.

##### a. WASHINGTON'S METHOD OF PRELIMINARY TREATMENT.

About 1 gram of rock powder is decomposed in a platinum dish or capacious crucible with dilute nitric and hydrofluoric acids. When all gritty particles have disappeared the solution is evaporated to dryness and reevaporated with nitric acid two or three times to

decompose fluorides and silicofluorides to a considerable extent.<sup>a</sup> The residue is treated on the bath with dilute nitric acid and eventually filtered from any undissolved matter. This last should be for precaution's sake always ignited, fused with a little sodium carbonate, extracted with water, the extract acidified with nitric acid and added to the main solution. (For further treatment see c, below.)

#### b. ALTERNATIVE METHOD OF PRELIMINARY TREATMENT.

Fuse with sodium carbonate, separate silica by a single evaporation with nitric acid, treat the ignited silica with hydrofluoric and nitric acids, evaporate to expel hydrofluoric acid, repeat the evaporation with nitric acid alone two or three times, bring the small residue into solution by boiling with nitric acid and add it to the main portion.

#### c. SUBSEQUENT TREATMENT.

Add to the solution obtained in a or b ammonium nitrate and molybdate solution as ordinarily prescribed.

The turbidity often observed on dissolving the precipitated and washed phosphomolybdate in ammonia is due to a compound of phosphorus. If the addition of a small fragment of a crystal of citric or tartaric acid fails to dissolve it, this should always be re-fused with sodium carbonate, extracted with water, and the filtrate otherwise treated as above, in order to secure the phosphorus in it.

According to F. A. Gooch and M. Austin,<sup>b</sup> in order to secure a magnesium-ammonium phosphate of normal composition, the procedure at this point should be as follows: To the phosphate solution, containing not more than 5 to 10 per cent of ammonium chloride and a slight excess of magnesia mixture, a little ammonia is added, and the precipitate is washed in due time with weak ammonia water. In general, however, as these conditions can seldom be fulfilled, they recommend to decant the supernatant liquid through the filter which is later to receive the precipitate, to dissolve this in as little hydrochloric acid as possible, to reprecipitate by dilute ammonia without further addition of magnesia mixture, and to wash finally with weakly ammoniacal water. Excess of ammonia, of ammonium salts, and of precipitant are all objectionable. In rock analysis the second precipitation will seldom be necessary. (For ignition, etc., of the precipitate, see this subject under "Magnesium," 11. B. a, p. 108.)

#### B. PROCEDURE WHEN MATERIAL IS SCANTY.

The following procedure admits of determining in the same portion, besides phosphorus, barium, iron, vanadium, chromium, and titanium,

<sup>a</sup> The method as originally described in Washington's Manual of the Chemical Analysis of Rocks has been modified by Doctor Washington and myself independently to the extent of doing away with the filtration of the precipitate of silicofluorides and fluorides before proceeding with the evaporation.

<sup>b</sup> *Am. Jour. Sci.*, 4th ser., vol. 7, 1899, p. 187; *Zeitschr. anorg. Chemie*, vol. 20, 1899, p. 121; *Chem. News*, vol. 79, 1899, pp. 233, 244, 255.

the last two either colorimetrically or gravimetrically, and is in large part extracted from a paper by Dr. T. M. Chatard.<sup>a</sup>

Silica is removed by hydrofluoric and sulphuric acids, excess of fluorine expelled, the residue brought into solution as far as possible with sulphuric or hydrochloric acid and hot water, filtered, the residue ignited, fused with sodium carbonate, dissolved in hydrochloric acid, and the solution, after precipitation of barium, added to the main one, which is now precipitated by ammonia to get rid of the magnesium salts usually present and thus insure a cleaner subsequent fusion with sodium carbonate.

The precipitated  $\text{Al}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{TiO}_2$  are dissolved in hot hydrochloric acid and filtered into a large platinum crucible, the filter burned and added, the solution evaporated to pastiness, a little water added to dissolve the salts, and dry sodium carbonate added in portions and stirred in thoroughly to prevent lumpiness in the fusion to follow, which is continued for half an hour. Addition of sodium nitrate is not necessary.

The fused mass is boiled out with water and washed with very dilute sodium-carbonate solution. In the residue iron and titanium can be determined by the methods already described. In the filtrate chromium can be determined colorimetrically if present in sufficient amount to give a pronounced color (see 17. B, p. 124). Afterwards, or immediately if the chromium is not to be thus estimated, rough ammonium nitrate is added to react with all the carbonate and the solution is digested on the bath till most of the ammonium carbonate is gone. Nearly if not quite all alumina is thus thrown out, carrying with it all phosphorus. The precipitate is washed with dilute ammonium-nitrate solution till the yellow color wholly disappears, after which it is dissolved in nitric acid and the phosphorus thrown out by molybdate solution. The filtrate from the aluminium and phosphorus, containing chromium and vanadium, can be treated as detailed in the following sections.

#### 17. CHROMIUM.

If vanadium is absent, or nearly so, as is apt to be the case in those highly magnesian rocks (peridotites), which usually carry a good deal of chromium, the following separation and gravimetric method for chromium gives good and concordant results, but in the presence of vanadium, and it is best generally to assume its presence, the colorimetric method should always be adopted.

<sup>a</sup> Am. Chem. Jour., vol. 13, 1891, p. 106; Bull. U. S. Geol. Survey No. 78, 1891, p. 87; Chem. News, vol. 63, 1891, p. 267.



## A. GRAVIMETRIC METHOD.

Having obtained chromium in solution as chromate and free from all else but a little alumina, as at the conclusion of the preceding section on phosphorus, proceed as follows:

Concentrate if necessary and add fresh ammonium sulphide, or introduce hydrogen sulphide. The chromium is reduced and appears as a precipitate of sesquioxide mixed with the rest of the alumina. This precipitate is now treated according to H. Baubigny<sup>a</sup> by dissolving in nitric acid, evaporating nearly to dryness, and heating with strong nitric acid and potassium chlorate, finally evaporating to dryness to get rid of the acid. Oxidation is complete and very speedy. On dilution with cold water, acid sodium carbonate is added in slight excess, and after two or three hours the precipitated alumina is filtered off. From the filtrate the chromium is then thrown out by fresh ammonium sulphide, redissolved, reprecipitated to free from alkali, and weighed.

The separation of aluminum from chromium by hydrogen peroxide in ammoniacal solution, as recommended by P. Jannasch and E. v. Cloedt,<sup>b</sup> has been shown by C. Friedheim and E. Brühl<sup>c</sup> to be valueless.

## B. COLORIMETRIC METHOD.

For this very accurate and by far the quickest method<sup>d</sup> for determining chromium in rocks and ores where the amount does not exceed a few per cent, there is needed the aqueous extract of a sodium-carbonate fusion of the rock (as obtained, for instance, under "Phosphorus," B, p. 122) in order to compare its color with that of a standard solution.

*α. Preparation and strength of standard solution.*—This standard solution is made by dissolving 0.25525 gram or double that amount of pure potassium monochromate in 1 liter of water made alkaline by a little sodium carbonate. Each cubic centimeter then corresponds to 0.1 or 0.2 milligram of chromic oxide ( $\text{Cr}_2\text{O}_3$ ), in which condition chromium is usually reported in rocks and ores. It is probably inadmissible to increase the strength of the standard much above the figure given.

*β. Preparation of the test solution.*—Before filtering the aqueous extract of the sodium-carbonate fusion a few drops of alcohol (ethyl or methyl) are added to destroy the color of sodium manganate. If the yellow color of the filtrate is very faint, concentration by evaporation will strengthen it, and less than 2 milligrams of chromic

<sup>a</sup> Bull. Soc. chimique, n. s., vol. 42, 1884, p. 291; Chem. News, vol. 50, 1885, p. 18.

<sup>b</sup> Zeitschr. anorg. Chemie, vol. 10, 1895, p. 402.

<sup>c</sup> Zeitschr. anal. Chemie, vol. 38, 1899, p. 681.

<sup>d</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 20, 1898, p. 454; Chem. News, vol. 78, 1898, pp. 227, 228; Geol. Survey No. 167, 1900, p. 37. First applied by L. de Koningh (Nederl. Tyds. voor de scheikunde) to the estimation of chromium in foodstuffs.

xide in 1 gram of rock can then be exactly measured. For smaller mounts it is best to employ from 3 to 5 grams of powder and then concentrate the chromium by precipitation by mercurous nitrate, as detailed in the next section under "Vanadium" (18. C, p. 127); otherwise it may be difficult or impossible, because of the large amount of alkali carbonate present, to obtain a filtrate of sufficiently small bulk to show a decided color.

If niter has been used in the fusion and the crucible has been at all attacked by it, a yellow coloration of the filtrate may be due to dissolved platinum, but neither the proportion of niter nor the temperature of the blast should ever be high enough to permit the crucible to be attacked. A passing yellowish coloration of the filtrate, due to this or some other cause, is not to be ascribed to chromium.

*γ. Comparison of colors.*—The final solution is transferred to a graduated flask of such size that its color shall be weaker than that of the standard chromium solution. Definite amounts of the latter are then diluted with water from a burette until of the same strength as the test solution, exactly as described on page 32. For very minute amounts it may be necessary to use Nessler tubes, as in ammonia estimations, instead of the glasses and apparatus there described and depicted.

As with colorimetric methods in general, this one gives better results with small than with large percentages of chromium, yet it can be applied in the latter cases with satisfactory results by making larger number of consecutive comparisons with the same solution.<sup>a</sup>

#### C. A FEW COMPARATIVE DATA.

A few comparisons between colorimetric and gravimetric determinations of chromium are here given to show the order of agreement, the former having been made several months and even years after the latter.

*Percentage determinations of chromium ( $\text{Cr}_2\text{O}_3$ ).*

Gravimetric.	Colorimetric.
Trace.	0.018
0.05	.051
.14	.12
.08	.083
Trace.	.013
None.	.0086
None.	.0067

The outcome was somewhat surprising, for it was hardly to be expected that the long and laborious quantitative separations should have resulted so well. It should be mentioned that for the gravi-

<sup>a</sup> D. W. Horn has studied the "Variable sensitiveness in the colorimetry of chromium." See *Am. chem. Jour.*, vol. 35, 1906, p. 253.

metric tests but 1 or 2 grams at most were used, which accounts for the reported absence of chromium in two instances, this report being based on the lack of color in the aqueous extract of the alkali fusion after removal of manganese.

## 18. VANADIUM (CHROMIUM) AND MOLYBDENUM.

### A. DISTRIBUTION OF VANADIUM AND MOLYBDENUM.

The wide distribution of vanadium throughout the earth's crust has in recent years been clearly established (see pp. 18-19), not only in ores and in coals, but in clays, limestones, sandstones, and igneous rocks.<sup>a</sup> I have shown<sup>b</sup> that vanadium occurs in appreciable amounts in the more basic igneous and metamorphic rocks up to 0.08 per cent or more of  $V_2O_5$ , but that it seems to be absent or nearly so from the highly siliceous ones. Some of their ferric aluminous silicate constituents carry still higher percentages—up to 0.13 per cent  $V_2O_5$  in a biotite separated from a pyroxenic gneiss. Molybdenum, on the other hand, appears to be confined in quantities susceptible of detection to the more siliceous rocks, and, except perhaps in rare instances, is not present in them in quantitatively determinable amount when operating on 5 grams of material. Hence the quantitative search for vanadium will usually be limited to rocks with less than 60 per cent of silica. The search for it even then will perhaps not often warrant the necessary expenditure of time, but in this connection it is to be remembered that neglect to determine it introduces an error in the figures for both ferrous and ferric oxides, which in extreme cases may be of considerable moment (see 7. B. b, p. 92; 19. C. c.  $\beta$ , p. 140).

### B. CONDITION OF VANADIUM IN ROCKS.

The connection below and elsewhere mentioned of vanadium with the ferric aluminous silicates of rocks, taken in connection with the existence of the mineral roscoelite, classed as a vanadium mica, indicates a condition of the vanadium corresponding to aluminum and ferric iron, and that it is to be regarded as replacing one or both of these elements. Hence it should be reported as  $V_2O_5$  and not as  $V_2O_3$ .

What its condition may be in matter of secondary origin, like clays, limestones, sandstones, coals, and ores of iron, is yet open to discussion. It was my opinion at one time that it should be regarded as in the pentavalent state ( $V_2O_5$ ), but my work on certain remarkable vanadiferous sandstones<sup>c</sup> of western Colorado, in which it unquestionably

<sup>a</sup> Hillebrand, W. F., Distribution and quantitative occurrence of vanadium and molybdenum in rocks of the United States: Am. Jour. Sci., 4th ser., vol. 6, 1898, p. 209; Chem. News, vol. 78, 1898, p. 216; Bull. U. S. Geol. Survey No. 167, 1900, p. 49.

<sup>b</sup> Loc. cit.

<sup>c</sup> Hillebrand, W. F., and Ransome, F. L., Am. Jour. Sci., 4th ser., vol. 10, 1900, p. 120.

occurs as trivalent vanadium ( $V_2O_3$ ), has led to a decided unsettling of this view. It is but proper to recall that C. Czudnowicz,<sup>a</sup> because of the extreme difficulty in completely extracting it from iron ores by an alkali-carbonate fusion and because of the easy reducibility of vanadic acid by ferrous salts, under the conditions in which brown iron ores are supposed to form, considered the vanadium in such ores to be in a lower condition of oxidation ( $V_2O_3$ ). O. Lindemann's<sup>b</sup> contrary conclusion with regard to certain iron ores, because the vanadium was extracted as  $V_2O_5$  by sodium-carbonate fusion without niter, is not valid, since this would probably be the case even if it existed in the ore as  $V_2O_3$ .

### C. AUTHOR'S METHOD.

#### a. DESCRIPTION OF THE METHOD.

In the following method there is nothing absolutely novel except that chromium and vanadium, when together, need not be separated, but are determined, the former colorimetrically, as already described (17. B., p. 124), the latter volumetrically, in the same solution. <sup>c</sup>

Five grams of the rock are thoroughly fused over the blast with 20 of sodium carbonate and 3 of sodium nitrate. After extracting with water and reducing manganese with alcohol it is probably quite unnecessary, if the fusion has been thorough, to remelt the residue as above, though for some magnetites and other ores containing larger amounts of vanadium than the generality of rocks, this may be necessary, as Edo Claassen has shown.<sup>d</sup> The aqueous extract is next nearly neutralized by nitric acid, the amount to be used having been conveniently ascertained by a blank test with exactly 20 grams of sodium carbonate, etc., and the solution is evaporated to approximate dryness. Care should be taken to avoid overrunning neutrality, because of the reducing action of the nitrous acid set free from the nitrite produced during fusion, but when chromium is present it has been my experience that some of this will invariably be retained by the precipitated silica and alumina, though only in one case have I observed a retention of vanadium, it being then large. The use of ammonium nitrate, instead of nitric acid, for converting the sodium carbonate into nitrate does not seem to lessen the amount of chromium retained by the silica and alumina.

As a precautionary measure, therefore, and always when chromium is to be estimated also, the silica and alumina precipitate should be evaporated with hydrofluoric and sulphuric acids, the residue fused with a little sodium carbonate and the aqueous extract again nearly neutralized with nitric acid and boiled for a few moments, the filtrate being added to the main one.

<sup>a</sup> Pogg. Ann., vol. 120, 1863, p. 20.

<sup>b</sup> Dissertation, Jena, 1878, thru Zeitsch. anal. Chemie, vol. 18, 1879, p. 99.

<sup>c</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 20, 1898, p. 461; Chem. News, vol. 78, 1898, p. 295; Bull. U. S. Geol. Survey No. 167, 1900, p. 44.

<sup>d</sup> Am. Chem. Jour., vol. 8, 1886, p. 437.

Mercurous nitrate is now added to the cold alkaline solution in some quantity, so as to obtain a precipitate of considerable bulk, containing, besides mercurous carbonate, chromium, vanadium, molybdenum, tungsten, phosphorus, and arsenic, should all happen to be in the rock. The mercurous carbonate serves to counteract any acidity resulting from the decomposition of the mercurous nitrate. Precipitating in a slightly alkaline instead of a neutral solution renders the addition of precipitated mercuric oxide unnecessary for correcting this acidity. If the alkalinity, as shown by the formation of an unduly large precipitate, should have been too great, it may be reduced by careful addition of nitric acid until an added drop of mercurous nitrate no longer produces a cloud.

After heating and filtering, the precipitate is ignited in a platinum crucible after drying and removing from the paper to obviate any chance of loss of molybdenum and of injury to the crucible by reduction of arsenic. The residue is fused with a very little sodium carbonate, leached with water, and the solution, if colored yellow, filtered into a graduated flask of 25 cm.<sup>3</sup> or more capacity. The chromium is then determined accurately in a few minutes by comparing with a standard alkaline solution of potassium monochromate (17. B, p. 124). Then, or earlier in absence of chromium, sulphuric acid is added in slight excess, and molybdenum and arsenic, together with occasional traces of platinum, are precipitated by hydrogen sulphide, preferably in a small pressure bottle.<sup>a</sup> If the color of the precipitate indicates absence of arsenic the filter with its contents is carefully ignited in porcelain, and the delicate sulphuric-acid test for molybdenum is applied as follows: The molybdenum compound is heated in porcelain with a single drop of strong sulphuric acid till the acid is nearly volatilized. On cooling a beautiful blue color is proof of the presence of molybdenum.

The filtrate, in bulk from 25 cm.<sup>3</sup> to 100 cm.<sup>3</sup>, is boiled to expel hydrogen sulphide, and titrated at a temperature of 70° to 80° with a very dilute solution of permanganate, representing about 1 milligram of V<sub>2</sub>O<sub>5</sub> per cubic centimeter, as calculated from the iron strength of the permanganate, one molecule of V<sub>2</sub>O<sub>5</sub> being indicated for each one of Fe<sub>2</sub>O<sub>3</sub>. One or two checks are always to be made by reducing again, by means of a current of sulphur-dioxide gas, boiling this out again,<sup>b</sup> and repeating the titration. The latter results are apt to be a very little lower than the first and are to be taken as the correct ones.

<sup>a</sup> From a sulphuric solution the separation of platinum and molybdenum by hydrogen sulphide is much more rapid and satisfactory than from a hydrochloric solution.

<sup>b</sup> The direct use of a solution of sulphur dioxide or of an alkali sulphite is inadmissible unless these have been freshly prepared, since after a lapse of time they contain other oxidizable bodies than sulphurous acid or a sulphite. The sulphur dioxide is best obtained as wanted by heating a flask containing a solution of sulphur dioxide, or of a sulphite to which sulphuric acid has been added.

The expulsion of the last traces of sulphur dioxide is said to be more effectively accomplished by boiling with simultaneous passage of a rapid current of carbon dioxide for a few minutes at the last than by boiling alone. Because of the small amount of air carried with it, long passage of the gas is said to result in slight oxidation of the vanadium (Manasse, O., *Ann. Chem. u. Pharm.*, vol. 240, 1887, p. 23; *Zeitschr. anal. Chemie* vol. 32, 1893, p. 225.)

## b. CONFIRMATORY QUALITATIVE TESTS.

In case the volume of permanganate used is so small as to make doubtful the presence of vanadium, it is necessary to apply a qualitative test, which is best made as follows: The solution is evaporated and heated to expel excess of sulphuric acid, the residue is taken up with 2 or 3 cm.<sup>3</sup> of water and a few drops of dilute nitric acid, and a couple of drops of hydrogen peroxide are added. A characteristic brownish tint indicates vanadium. Unless the greater part of the free sulphuric acid has been removed the appearance of this color is sometimes not immediate and pronounced, hence the above precaution. It is also necessary that the nitric acid shall be in considerable excess, since in a neutral or only faintly acid solution the color does not appear strongly.

The above is a surer test to apply than the following: Reduce the bulk to about 10 cm.<sup>3</sup>, add ammonia in excess, and introduce hydrogen sulphide to saturation. The beautiful cherry-red color of vanadium in ammonium-sulphide solution is much more intense than that caused by hydrogen peroxide in acid solution, but the action of ammonia is to precipitate part or all of the vanadium with the chromium or aluminum that may be present or with the manganese used in titrating, and ammonium sulphide is unable to extract the vanadium wholly from these combinations. Usually, however, the solution will show some coloration, and addition of an acid precipitates brown vanadium sulphide, which can be collected, ignited, and further tested if desired.

## c. APPLICATION OF THE METHOD IN PRESENCE OF RELATIVELY MUCH CHROMIUM.

The application of the method in its foregoing simplest form is subject to one limitation—the chromium must not be present above a certain moderate amount. This limitation is due to the considerable amount of permanganate then required to produce a clear transition tint when titrating in a hot solution, as is advisable with vanadium. In a cold solution of chromic sulphate much less permanganate is needed to produce the peculiar blackish tint without a shade of green, which affords a sure indication of excess of permanganate, but in a hot and especially a boiling solution the oxidation of the chromium itself takes place so rapidly that a very large excess of the reagent may be added before a pronounced end reaction is obtained. Nevertheless, fairly satisfactory determinations of as little as 1 or 2 milligrams of vanadium pentoxide can be made in presence of as much as 30 milligrams of chromic oxide. To accomplish this it is only necessary to apply a simple correction obtained by adding permanganate to a like bulk of equally hot chromic sulphate solution containing approximately the same amount of chromium.

C. H. Ridsdale<sup>a</sup> titrated the cold solution to avoid oxidation of chromium and obtained accurate results, but in my experience the end reaction is then uncertain.

The following tables contain the results of a considerable number of tests, those in Table 2 being tabulated separately in order to show the degree of accuracy attainable with a large excess of chromium by applying the correction mentioned above and also the amount of this correction:

TABLE 1.—*Tests for vanadium in the presence of chromium.*

No.	Chromic oxide.	Vanadium pentoxide.	Vanadium pentoxide found.	Error.	No.	Chromic oxide.	Vanadium pentoxide.	Vanadium pentoxide found.	Error.
	<i>Milligrams</i>	<i>Milligrams</i>	<i>Milligrams</i>	<i>Mg.</i>		<i>Milligrams</i>	<i>Milligrams</i>	<i>Milligrams</i>	<i>Mg.</i>
1	1	9.87	9.22	—0.15	7	3.5	18.74	18.97	+ .23
2	1	.94	1.04	+ .10	8	6	5.6	6.1	+ .50
			.98	+ .04	9	6	4.68	4.78	+ .10
3	1.5	5.25	5.49	+ .24	10	6	5.62	5.58	— .04
			5.43	+ .19	11	10	5.62	5.58	— .04
4	2	5.62	5.5	— .12	12	10	23.52	23.81	+ .29
			5.5	— .12				23.71	+ .19
5	3	4.68	4.78	+ .10	13	10	46.85	46.98	+ .13
			4.78	+ .10				47.20	+ .35
			4.83	+ .15	14	25	23.52	23.65	+ .13
6	3	5.62	5.58	— .04				23.75	+ .23
			5.58	— .04	15	87.5	23.52	23.71	+ .19
7	3.5	18.74	18.89	+ .15					

TABLE 2.—*Application of correction for larger amounts of chromium, obtained by adding potassium permanganate to an equal bulk of solution containing a like amount of chromic sulphate.*

No.	Chromic oxide.	Vanadium pentoxide.	Vanadium pentoxide found.		Error.	Volume of solution.
			Uncorrected.	Corrected.		
	<i>Milligrams.</i>	<i>Milligrams.</i>			<i>Milligram.</i>	
16	20	0.94	1.59	0.99	+0.05	50 to 100 cm. <sup>3</sup>
17	20	1.87	2.69	2.09	+ .22	50 to 100 cm. <sup>3</sup>
			2.39	1.79	— .08	
			2.59	1.99	+ .12	
18	20	18.74	19.4	18.73	— .01	50 to 100 cm. <sup>3</sup>
			19.3	18.63	— .11	
			19.3	18.63	— .11	
19	30	1.87	2.99	2.14	+ .27	About 100 cm. <sup>3</sup>
			2.79	1.94	+ .07	
			2.79	1.94	+ .07	
			2.69	1.84	— .03	
			2.69	1.84	— .03	
20	30	1.87	2.69	1.79	— .08	200 cm. <sup>3</sup>
			2.89	2.09	+ .22	
			2.89	2.09	+ .22	
			2.79	1.99	+ .12	
21	62	46.85	48.60	47.60	+ .75	200 cm. <sup>3</sup>

In spite of the fact that the correction in most of the trials of this last table represents a large proportion of the permanganate used, the results must be considered satisfactory in view of the small amount of vanadium present, and they show that the method in competent hands after a little experience affords trustworthy figures.

<sup>a</sup> Jour. Chem. Soc., vol. 7, 1888, p. 73.

The method of T. Fischer <sup>a</sup>—digestion of the precipitated lead salts with a strong solution of potassium carbonate—appears to offer the long-needed satisfactory quantitative separation of arsenic, phosphorus, chromium, tungsten, and molybdenum from vanadium, the normal lead metavanadate remaining quite unattacked, according to Fischer, while the other lead salts are wholly decomposed, but the applicability of this method to the separation of the minute amounts often found in rocks and ores has not been tested. The object has been in the present case to reach satisfactory results with the greatest expedition, and when chromium is not present in considerable amount this is accomplished.

Fortunately, chromium is almost never a prominent constituent of clays, coals, iron ores, and those rocks in which vanadium has thus far been reported, for although it is usually certain of the most basic of the silicate rocks that are highest in chromium—as the peridotites—yet in these, so far as present experience teaches, vanadium is lacking, a fact doubtless connected with the simultaneous absence from them of ferric aluminous silicates.

#### D. COLORIMETRIC METHOD FOR VANADIUM.

In the paper already cited <sup>b</sup> the possibility of a colorimetric method for vanadium, based on its behavior toward hydrogen peroxide, was suggested. L. Maillard <sup>c</sup> has since developed such a method, though it has not been tested in the Survey laboratory.

### 19. FERROUS IRON.

#### A. COMPARISON OF SEALED-TUBE AND HYDROFLUORIC-ACID METHODS—COMPARATIVE WORTHLESSNESS OF THE FORMER IN ROCK ANALYSIS.

No point in rock analysis has been the cause of greater solicitude to the chemist, and especially to the mineralogist and petrographer, than the determination of iron in ferrous condition. The sealed-tube or Mitscherlich method with sulphuric acid, for a long time the only available one, is in theory perfect, since complete exclusion of oxygen is easily attainable. Its earliest recognized defect lies in the inability to secure always complete decomposition of the iron-bearing minerals, and even to ascertain, oftentimes, whether or not the decomposition has been complete. The addition of hydrofluoric acid to the sulphuric in the tube, in order to insure this breaking up, is to be regarded as of very doubtful utility in most cases, since the glass may be so strongly attacked as to add an appreciable amount of iron to the

<sup>a</sup> Inaugural Dissertation, Rostock, 1894.

<sup>b</sup> Hillebrand, W. F., Jour. Am. Chem. Soc., vol. 20, 1898, p. 461; Chem. News, vol. 78, 1898, p. 295, Bull. U. S. Geol. Survey No. 167, 1900, p. 44.

<sup>c</sup> Bull. Soc. chim., vol. 23, 1900, p. 559.



solution, and the hydrofluoric acid may have exhausted itself in attacking the glass before the more refractory minerals succumb. Nevertheless, if decomposition can be effected by sulphuric acid alone the results obtained are sharp and concordant; but they are in rock analysis usually higher than when made by any of the modifications of the hydrofluoric-acid method now so extensively practised. This difference is not very marked with rocks containing but 1 or 2 per cent of ferrous iron, but it increases with rising percentage to such an extent that where the sealed-tube method will show 12 per cent ferrous oxide the other may indicate no more than 10 per cent. This is a fact of which I have long been cognizant, but it seems not to have been known to chemists or petrographers at large, though E. A. Wülfing<sup>a</sup> noticed this difference in certain analyses without appreciating its significance. Experiments with soluble iron salts of known composition, like ferrous sulphate and ferrous-ammonium sulphate, throw no light on the subject, for both methods give with them the same sharp and accurate results.

The key to the problem was discovered by L. L. de Koninck<sup>b</sup> a good many years ago, but remained unknown to the chemical world by reason of its obscure medium of publication until rediscovered by Dr. H. N. Stokes in this laboratory, during an investigation on the action of ferric salts on pyrite and other sulphides.<sup>c</sup> That oxidation of the sulphide and reduction of the ferric salt hereby takes place was recognized by J. H. L. Vogt,<sup>d</sup> but not the ease with which the change takes place and the completeness of the oxidation of the pyrite, not only of its iron but of the greater part of the sulphur as well. Pure pyrite itself is attacked with extreme slowness by boiling dilute sulphuric and hydrofluoric acids, either alone or mixed, but the moment a ferric salt is introduced the case is altogether different.

The complete solution to the problem was afforded by the observation made in the Survey laboratory that rocks with hardly an exception and many minerals carry pyrite or pyrrhotite, or both, often in considerable amount, often in traces only. Sulphur can almost always be detected in 2 grams of rock powder.

Experiment has shown (C. c.  $\alpha$ , p. 138) that with the amounts of sulphides usually found in igneous rocks their effect on the estimation of ferrous iron by the hydrofluoric-acid method at atmospheric pressure and boiling heat is negligible, though by increasing the amount of sulphide the effect becomes more and more apparent, because of the greater surface of pyrite exposed to the action of the ferric iron of the rock.

<sup>a</sup> Ber. Deutsch. chem. Gesell., vol. 32, 1899, p. 2217, footnote.

<sup>b</sup> Ann. Soc. géol. Belgique, vol. 10, 1882-83, p. 101; Zeitschr. anorg. Chemie, vol. 26, 1901, p. 123.

<sup>c</sup> Bull. U. S. Geol. Survey No. 186, 1901; Am. Jour. Sci., 4th ser., vol. 12, 1901, p. 414.

<sup>d</sup> Zeitschr. prakt. Geol., 1899, pp. 250-251.

Under the conditions of the Mitscherlich method, on the other hand—a temperature of 150 to 200°, and even higher, high pressure, much longer time of action, and impossibility of escape of any hydrogen sulphide that may be formed—the sulphur of the sulphides becomes nearly if not fully oxidized to sulphuric acid at the expense of the ferric iron in the rock, with the production of an equivalent amount of ferrous iron in addition to that resulting from the sulphide itself.

Let us now see what the effect of these traces of sulphides when fully oxidized amounts to. One atom of sulphur (32) requires for its complete conversion to trioxide the oxygen of three molecules of ferric oxide (480), which then become six molecules of ferrous oxide (432). In other words, 0.01 per cent of sulphur may cause the ferrous oxide to appear too high by 0.135 per cent, and 0.10 per cent of sulphur may bring about an error of 1.35 per cent in ferrous oxide. The case is still worse if the sulphur is set free as hydrogen sulphide from a soluble sulphide, for then the above percentages of sulphur produce errors of 0.18 and 1.8 per cent, respectively, in the ferrous oxide determination.

The error caused by sulphides tends to become greater the more there is present of either or both sulphide and ferric salt. Now, the highly ferruginous rocks usually carry more ferric iron than the less ferruginous ones, and they are often relatively high in pyrite and pyrrhotite; hence the increasing discrepancy between the results by the two methods as the iron contents of the rocks rise is fully in accord with the above explanation.<sup>a</sup>

Of course carbonaceous matter will, under the conditions of the Mitscherlich method, likewise reduce sulphuric acid and cause the determination of ferrous iron to be faulty.

Notwithstanding the fact that the Mitscherlich method has thus been discredited in its general applicability to rocks and minerals, it is still probably the best with those which are totally free from sulphides and wholly decomposable. Hence the conditions under which success can best be achieved by it are set forth in the following paragraphs.

#### B. THE MODIFIED MITSCHERLICH METHOD.

*α. Strength of acid.*—The method in its original and usual application calls for a mixture of 3 parts of sulphuric acid and 1 of water by weight, or about 3 to 2 by volume, though a still stronger acid is sometimes used. In some cases, however, perhaps in most, much better decomposition of the silicates is effected by reversing the proportions of water and acid, or at any rate by diluting considerably beyond the

<sup>a</sup> For details of experiments showing the worthlessness of the Mitscherlich method for rocks and minerals which contain even a trace of free sulphur or sulphides, see Hillebrand, W. F., and Stokes, H. N., *Relative value of the Mitscherlich and hydrofluoric-acid methods for ferrous-iron determinations*: Jour. Am. Chem. Soc., vol. 22, 1900, p. 625; and *Zeitschr. anorg. Chemie*, vol. 25, 1900, p. 326.

above proportion. Hereby the separation of salts difficultly soluble in the stronger acid is avoided and the actual solvent effect on the minerals seems to be in no wise diminished.

*β. Filling, sealing, and heating of the tube.*—The very finely powdered mineral having been introduced into a tube of resistant glass free from ferrous iron, the open end is drawn out, so as to leave a funnel for the introduction of the acid. A very little water is then introduced and carefully heated to boiling for a moment to expel all air from the powder. The diluted acid—which has just been boiled down from a state of greater dilution in order to have it free from air—is then poured in until the tube is about three-fourths filled. Carbon dioxide is then introduced, from a generator which has been in active operation for some time, through a narrow glass tube drawn out of the same kind of glass as that of which the decomposing tube consists. In a few moments the air is expelled, and the small tube is then sealed into the large one over the blast lamp without interrupting the gas current until the very last instant, when to prolong it would perhaps cause a blowing out of the softened glass. The interruption of the current at the proper moment is easily effected by the pressure of the thumb and finger holding the small tube at the point where it enters the rubber tube leading from the gas generator. No breakage in the oven ever occurs as a consequence of thus fusing one tube into the other.

The heating is done in a bomb oven at any desired temperature up to, say, 200°, and continued at intervals until examination by aid of a low-power lens shows that decomposition is complete or has progressed as far as can be hoped for. By inclosing the glass in an outer tube of strong steel, properly capped <sup>a</sup> and containing a little ether or benzine to equalize the pressure on both sides of the glass, the temperature can be elevated far beyond what is otherwise permissible, and the decomposition will then doubtless be more complete with refractory silicates.

*γ. Reason for introducing gas and sealing as above directed.*—The usual practise in employing the above method has been to expel air before sealing by introducing a few crystals or lumps of an alkali carbonate or bicarbonate, the gas set free on their contact with the acid being supposed to effectively expel all air. That this is not accomplished the following series of comparative results, long since published elsewhere, <sup>b</sup> fully shows. The material used was the oxide of uranium  $U_3O_8$ , requiring by theory 32.07 per cent of  $UO_2$ . Operating as just described on from 0.3 to 0.5 gram, the results were

31.06, 31.07, 29.72, 29.33, 29.89, 30.69,

<sup>a</sup> Ullmann, C., *Zeitschr. für angew. Chemie*, 1893, p. 274; *Zeitschr. anal. Chemie*, vol. 33, 1894, p. 582.

<sup>b</sup> *Bull. U. S. Geol. Survey* No. 78, 1891, p. 50; *Chem. News*, vol. 64, 1891, p. 232.

whereas after filling the tube with gas from a generator there was found

32.11, 31.90, 32.15, 32.12, 32.06, 32.17, 32.28,

the average error of the former series being 1.78 per cent. The percentage error would, of course, be reduced by increasing the weight of mineral operated on. An average error equal to the above when employing 1 gram of ferrous minerals would make the percentage for FeO about 0.3 per cent too low. While the absolute error might be the same in all cases, the relative error would increase with minerals low in ferrous iron.

### C. THE HYDROFLUORIC-ACID METHOD.

#### a. PRINCIPLE OF THE METHOD.

This method consists simply in securing complete decomposition of the very fine powder in absence of air by means of hydrofluoric acid in presence of sulphuric acid, and titrating the ferrous iron in the resulting solution. It is the one which has been almost exclusively used in the Survey laboratories. The form of apparatus hitherto employed is the original one devised by J. P. Cooke<sup>a</sup> (fig. 20, p. 141). This does not permit driving off the superfluous hydrofluoric acid, as does that of Treadwell (fig. 21, p. 143) to some extent, which latter ought therefore, for a reason developed below, to afford slightly lower and more accurate results, other conditions being equal. But the method as ordinarily carried out is subject to two defects of opposite sign but unequal value, the second and greater of which has remained unsuspected until recently.

It is possible to titrate ferrous iron in presence of sulphuric acid and as much as 5 to 7 cm.<sup>3</sup> of 40 per cent hydrofluoric acid in a total volume of 200 to 400 cm.<sup>3</sup> almost if not quite as exactly as in sulphuric acid alone, provided the iron solution is diluted with air-free water and the titration is made immediately after adding the hydrofluoric acid and with all possible dispatch. This condition can naturally not be fulfilled in practice because the mineral must be in contact with the acid for a long time in the apparatus commonly employed, and it seems to be almost impossible to prevent some oxidation during this period.

#### b. DEFECTS OF THE METHOD.

*α. Oxidizability of divalent manganese by permanganate in presence of hydrofluoric acid.*—Manganic fluoride in dilute solutions is hardly at all dissociated, wherein it differs from manganic sulphate. The entering of the manganic ion as fast as formed into the undissociated state explains the fact that in presence of hydrofluoric acid

<sup>a</sup> Am. Jour. Sci., 2d ser., vol. 44, 1867, p. 347.

divalent manganese is readily oxidized by permanganate, whereby a sharp end reaction is rendered unattainable in presence of much hydrofluoric acid, say over 7 cm.<sup>3</sup> of 40 per cent acid. It is easy to obtain a transitory pink color throughout the liquid, but this rapidly disappears, the more rapidly the greater the amount of hydrofluoric acid or of manganous salt present. The oxidizer can be added by the cubic centimeter to solutions already containing manganous sulphate in presence of hydrofluoric acid without producing a more than passing pink color. The solution, however, takes on in ever increasing intensity the red-brown color characteristic of manganic salts. The decolorization due to this cause is hence much more pronounced in the case of rocks high in ferrous iron than in that of those low in this constituent, because of the greater amount of manganous salt resulting from reduction of a correspondingly larger amount of permanganate, and is also greater with increasing hydrofluoric acid. In presence of but little ferrous iron, up to, say, 2 centigrams, and 5 to 7 cm.<sup>3</sup> of hydrofluoric acid the color produced by a drop of permanganate lasts some time, but is very evanescent as the ferrous iron, and consequently the manganous salt formed, increases.

E. Deussen,<sup>a</sup> on the basis of but a few tests, ascribes the extra consumption of permanganate to some obscure action of iron and seeks to counteract it by the addition of manganous sulphate. In both these assumptions he is, however, certainly in error, as shown by the fact that the addition of a drop or two of this oxidizer to a solution of ferric sulphate in even a large amount of hydrofluoric acid produces a very stable coloration, which is destroyed by the addition of manganous sulphate and by the above-mentioned very evident partial reoxidation of the reduced manganese. This last, the oxidation of divalent manganese by permanganate in presence of hydrofluoric acid, can be effected with the greatest ease in entire absence of ferric iron.

Numerous tests have been made to ascertain the error due to oxidation of the manganese under conditions similar in respect to the amounts of iron involved to those encountered in rock analysis. Increasing amounts of hydrofluoric acid were added to fixed amounts of ferrous sulphate and sulphuric acid and the titration made with the least possible expenditure of time. With not more than 7 cm.<sup>3</sup> of 40 per cent hydrofluoric acid a very slight tendency was observed toward high results, but in most cases the results were identical with those obtained in presence of sulphuric acid alone. As the amount of hydrofluoric acid was increased to 10 cm.<sup>3</sup> a perceptible increase was noticed, which became markedly greater with 15 cm.<sup>3</sup> of the acid, amounting in this case to about 0.2 cm.<sup>3</sup> in the average on a normal consumption of 20 cm.<sup>3</sup> of permanganate of 0.0032 FeO titer. The personal factor enters here, G. Steiger having found slightly greater

<sup>a</sup> Zeitschr. anorg. Chemie, vol. 44, 1905, p. 425.

differences, but it is possible that they resulted from his titrating in more concentrated solutions, for with greater dilution the differences are unquestionably less, according to experiments of my own.

Hence, after decomposition of the rock, removal of the excess of hydrofluoric acid is called for if the best results are to be expected. To a certain extent Treadwell's apparatus accomplishes this, but, as the experiments show, its complete expulsion is unnecessary.

*β. Extreme oxidizability of divalent iron by free oxygen in presence of hydrofluoric acid or a fluoride.*—In comparing two series of results obtained with the Cooke apparatus, using a solution of ferrous sulphate in sulphuric acid alone in the one and with hydrofluoric acid in addition in the other, a minus error was in all cases observed when the latter acid was present. An observation made by R. Peters,<sup>a</sup> but not applied by him to the present case, explains this. Ferric fluoride, like the corresponding manganic salt, is almost undissociated in solution, whereas the sulphate undergoes considerable dissociation, and in this state counteracts, to a great extent, the tendency of any ferrous iron present to become oxidized by free oxygen. Hence the slow oxidation of divalent iron by air in sulphuric-acid solution and its very rapid oxidation in presence of hydrofluoric acid. While a sulphuric solution of ferrous sulphate exposed to the air in an open dish will hardly change in strength during an hour, a similar solution to which hydrofluoric acid has been added will suffer a large measure of oxidation in a quarter of the time. The change in titer becomes pronounced after a few minutes.

In the experiments with the Cooke apparatus just referred to, the minus error should amount to not more than 0.1 to 0.2 cm.<sup>3</sup> of permanganate on a normal consumption of 20 cm.<sup>3</sup> or 0.5 to 1 per cent of the iron, but it may be considerably greater if the utmost care is not taken to employ carbon dioxide as free as possible from air and to exclude most carefully the entrance otherwise of air into the apparatus. It will also be greater the higher the concentration, within certain limits, of the solution during titration.

With a view to lessening the error, if possible, the mode of operating was varied by doing away with carbon dioxide, except at the start, shutting it off as soon as the bath was in active ebullition, and transferring the crucible direct from the hot bath to the titration vessel. The results were, if anything, a trifle better. As the employment of either the Cooke or the Treadwell apparatus involves long contact of the mineral with the acids, and as the experiments last mentioned were favorable in their results, it seemed as if the simple method of J. H. Pratt<sup>b</sup> might be modified in the same sense. He avoided the use of all apparatus other than a capacious platinum crucible fitted with a perfo-

<sup>a</sup> *Zeitschr. phys. Chemie*, vol. 26, 1898, p. 193.

<sup>b</sup> *Am. Jour. Sci.*, 3d ser., vol. 48, 1894, p. 149.

rated cover for the introduction of carbon dioxide, in which he boiled the mineral powder with sulphuric and hydrofluoric acids, the decomposition being greatly hastened by the active movement and higher temperature and usually completed in five to ten minutes. The modification consists in doing away altogether with carbon dioxide, except at the start, and depending on the steam of the boiling iron solution to exclude air, a modification which Pratt himself tried with rather considerable minus errors, resulting perhaps from his having a smaller crucible at command than the method really demands. With a crucible of 100 cm.<sup>3</sup> capacity, the following results with ferrous sulphate show what are the possibilities of the method:

*Ferrous-iron determinations by the modified Pratt method.*

[Strength of permanganate 0.0032 FeO per cm.<sup>3</sup>]

Time of boiling.	Permanganate used.	Normal consumption of permanganate	Time of boiling.	Permanganate used.	Normal consumption of permanganate.
<i>Minutes.</i>	<i>cm.<sup>3</sup></i>	<i>cm.<sup>3</sup></i>	<i>Minutes.</i>	<i>cm.<sup>3</sup></i>	<i>cm.<sup>3</sup></i>
10	4.9	4.8	10	19.4	19.2
10	4.8	4.8	10	19.3	19.2
10	5.0	4.8	15	19.25	19.2
10	4.9	4.8	15	19.1	19.2
10	9.6	9.6	15	19.3	19.2
10	9.6	9.6	20	19.2	19.2
10	9.6	9.6	20	19.3	19.2
10	19.2	19.2			

These results leave little, if anything, to be desired. They show either normal values or a slight plus error instead of the invariable negative one of all previous determinations. The method has not yet been tested on rocks in comparison with that of Cooke, so that it would be premature to assert that this is the one to employ in preference to the other or to that of Treadwell, but the indications are so favorable that extended tests will be carried out in the regular course of Survey work.

C. INFLUENCE OF SULPHIDES, VANADIUM, AND CARBONACEOUS MATTER ON THE DETERMINATION OF FERROUS IRON BY THE HYDROFLUORIC-ACID METHOD.

A dark color of the undissolved residue may be due to pyrite, graphite, or carbonaceous matter. The first of these affects the result but little, the second probably not at all, and they can be distinguished by their behavior toward nitric acid. Organic matter of course renders impossible the determination of ferrous iron.

*α. Sulphides.*—Pyrite, in the quantities usually met with in igneous rocks, is probably without serious effect on the ferrous-iron determination by any of the hydrofluoric-acid methods. This sulphide is very resistant toward attack in the absence of oxygen, as is shown by the fact that if present in any quantity it can be readily recognized in the residue after titration. In any case it is impossible to allow for an

error introduced by its possible decomposition, and the result of titration must count as ferrous iron. In the case of soluble sulphides two sources of error are introduced—that of reduction of ferric iron by hydrogen sulphide evolved, and that due to the ferrous iron which the sulphides themselves may contain, especially if pyrrhotite is present. The first of these is perhaps negligible, since most of the hydrogen sulphide would probably be expelled without reducing iron. The second is approximately measurable if it is known that pyrrhotite is the only soluble sulphide present, and its amount has been ascertained by determining the hydrogen sulphide set free on boiling with hydrochloric acid in a current of carbon dioxide. In this case a correction is to be applied to the result of titration for total ferrous iron (see also 24. B. b, p. 160, under "Sulphur").

In order to obtain quantitative data regarding the effect of pyrite on the ferrous-iron estimation by the hydrofluoric-acid method the following tests were made: Part of a fine crystal of pyrite was rather finely powdered and boiled with dilute sulphuric acid, which extracted considerable ferrous iron, derived presumably from admixed or intergrown pyrrhotite, since a second boiling with fresh acid gave a negative test for ferrous iron. After washing by decantation with water, followed by alcohol and ether, the powder was dried and further pulverized. A quarter of a gram of it when treated with hydrofluoric and sulphuric acids in a large crucible by the Cooke method for ferrous iron, then rapidly filtered through a very large perforated platinum cone fitted with filter paper, required but 2 drops of a permanganate solution representing only 0.0032 gram FeO to the cubic centimeter.

Since, however, Dr. H. N. Stokes has found<sup>a</sup> that the oxidizing effect of ferric salts on pyrite and other sulphides is vastly greater than seems to have been suspected (see p. 132), the following tests were made in order to ascertain the probable error due to this action under the conditions prevailing in rock analysis: Successive portions of 1 gram each of a horn blende schist, free from sulphur and carrying 10.09 per cent FeO as the mean of several determinations and 4.00 per cent  $\text{Fe}_2\text{O}_3$ , were mixed in a large 100 cm.<sup>3</sup> platinum crucible with 0.02, 0.025, and 0.10 gram, respectively, of the above purified pyrite powder, and treated with hydrofluoric and sulphuric acids by the Cooke method, the water bath being at boiling heat for one hour. The cooled contents of the crucible were poured into a platinum dish containing water and titrated rapidly nearly to an end. Then, in order to get rid of the pyrite, which would obscure the end reaction by its reducing effect on the permanganate, the solution was filtered as above and in the clear filtrate the titration was carried to completion. The results were 10.02, 10.16, and 10.70. Inasmuch as the smallest of these three charges of

<sup>a</sup> Bull. U. S. Geol. Survey No. 186, 1901; Am. Jour. Sci., 4th ser., vol. 12, 1901, p. 414.



pyrite was several times greater than what may be considered an unusually high amount for an igneous rock, it is very evident that for all practical purposes the influence of pyrite on the ferrous determination by the Cooke method is negligible. At the same time it is to be borne in mind that with increased content in ferric iron an increased amount of pyrite will be attacked, and that the extent of this attack is influenced by the degree of fineness of the pyrite powder.

*β. Vanadium.*—If vanadium, when present, exists in the trivalent condition, it necessarily affects with an error varying with its amount the result of titration for ferrous iron. If the amount of vanadium is known a correction can be applied as follows: One molecule of  $V_2O_3$  (150.8) in oxidizing to  $V_2O_5$  requires as much oxygen as four molecules of FeO (288) when oxidized to  $Fe_2O_3$ . The proportion, 150.8 : 288 ::  $V_2O_3$  present :  $x$ , therefore gives the figure to be deducted from the uncorrected value for FeO. That this correction is very needful with many of the basic rocks becomes at once evident from the following perhaps extreme example:

Found 2.50 per cent apparent FeO in a rock containing .13 per cent  $V_2O_3$ .

Deduct .25 per cent FeO equivalent in its action on  $KMnO_4$  to .13  $V_2O_3$ .

Leaving 2.25 per cent FeO corrected.

Found 5.00 per cent apparent total iron as  $Fe_2O_3$  in the same rock.

Deduct .14 per cent  $Fe_2O_3$  corresponding to .13 per cent  $V_2O_3$ .

Leaving 4.86 per cent corrected total iron as  $Fe_2O_3$ .

Deduct 2.50 per cent  $Fe_2O_3$  equivalent to 2.25 per cent FeO.

Leaving 2.36 per cent  $Fe_2O_3$  in the rock.

Failure to correct for the vanadium in both cases would have made the figures for FeO and  $Fe_2O_3$ , respectively, 2.50 and 2.22 instead of 2.25 and 2.36 as shown above.

*γ. Carbonaceous matter.*—As said before (c, p. 138), matter of organic origin other than graphitic carbon renders the results of the ferrous-iron determination altogether unreliable.

#### d. THE METHOD IN ITS VARIOUS MODIFICATIONS.

*α. According to Cooke.*<sup>a</sup>—The apparatus, as shown in fig. 20, consists of a small water bath of a single opening and covered with a glass funnel the stem of which has been cut off near the flare, resting in a troughlike depression of the specially made cover. Into this trough water constantly drops from a tubulated bottle, thus securing a perfect water joint and serving to keep the bath full by overflowing on the inside. For the more perfect exclusion of air it is best to use water that has been freshly boiled. Through a small metal pipe carbonic acid gas flows into the bath under the cover, but above the surface of the water, and rising through notches in the edge of the opening of the cover fills the funnel and crucible. (See footnote<sup>b</sup>, p. 91.)

<sup>a</sup> Am. Jour. Sci., 2d ser., vol. 44, 1867, p. 347.

A gram of the finely ground powder<sup>a</sup> is placed in a platinum crucible of 80 to 100 cm.<sup>3</sup> capacity and stirred up therein with water enough to moisten and about 10 cm.<sup>3</sup> of dilute sulphuric acid (1 acid to 4 to 6 water by volume). If the rock contains carbonates there will be effervescence, hence the acid must be added cautiously and the

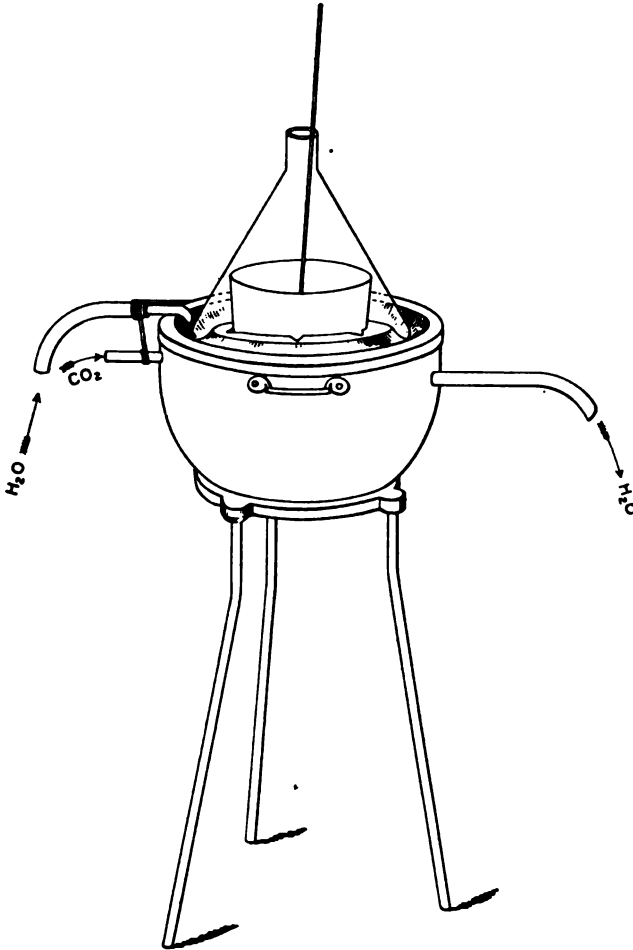


FIG. 20.—Cooke's apparatus for ferrous-iron determination.

cover placed on till action is over. There need be no fear of oxidation of ferrous iron at this stage, should any go into solution. The cover is rinsed if need be and the crucible at once placed in the opening of the bath, the funnel put in place, the gas current started, as also the flow of water from the reservoir, and the lamp under the bath already

<sup>a</sup> The fineness of grinding will depend on the nature of the rock. Most granitic rocks require but moderately fine grinding, those high in ferruginous and refractory minerals generally, as tourmaline, need to be very fine.

full of water is lit. As soon as it can be safely assumed that the air in both funnel and crucible has been displaced a measured volume of strong hydrofluoric acid (5 to 7 cm.<sup>3</sup>) is poured into the crucible through a funnel of platinum or rubber and a platinum stirring rod is inserted through the stem of the glass funnel into the crucible. In absence of a suitable funnel for the acid, the glass may be momentarily raised for the introduction of the acid, but not before the crucible has become filled with carbon dioxide.

When steam issues strongly from the funnel, the gas current from the generator is stopped and the apparatus left to itself for an hour, except for occasional stirring of the powder. When no gritty matter can longer be felt or when experience tells that decomposition must be complete, the gas current is again turned on, the lamp extinguished, and the water flow increased to the capacity of the outlet tube from the bath. In fifteen minutes the crucible and contents should be cool. The rod is then removed and placed in the titration vessel containing freshly boiled water (usually about 300 cm.<sup>3</sup>), which is already under the burette, the contents of the crucible quickly follow with the rinsings, and the permanganate is run in as rapidly as possible with constant but gentle stirring till the first pink blush appears throughout the whole liquid for at least a couple of seconds. With little iron the color will last some time, but with increasing amounts, as also with increasing amounts of hydrofluoric acid, it fades out with ever greater rapidity. Until the operator has become experienced a duplicate determination should always be made. It is to be noted that the crucible is always much fuller at the end of the heating than when first placed on the bath, because of condensation of steam.

Instead of allowing the crucible to cool in a current of carbon dioxide, it may be at once transferred to the titration dish and permanganate run in as above directed. If carefully done, the experiments already referred to seem to show that the results by this variation of the method may be a trifle better than those obtained after cooling.

Those inexperienced with the method in either variation should not fail to test it first with solution of ferrous sulphate that has been standardized without hydrofluoric acid, and in practice duplicate determinations should be made if possible.

*β. According to Pratt (modified).*—One gram of the finely ground powder is treated in a capacious crucible, as in *α*, with water enough to moisten and about 10 cm.<sup>3</sup> of dilute sulphuric acid, and the crucible with cover on is placed on a triangle well down over a lamp turned low and protected from drafts. The air in the crucible is displaced rapidly by carbon dioxide entering beneath the lid slightly raised on one side. In a few seconds the liquid boils, but before allowing this to happen the gas current is stopped and the well-fitting lid lowered.

Then from a small platinum crucible a measured quantity of strong hydrofluoric acid (5 to 7 cm.<sup>3</sup>) is poured in with one hand, while the other draws the lid a little to one side. A second suffices. The lid is replaced, the flame is increased for a few moments with great caution not to allow boiling over, and the moment steam is seen to issue around the lid it is again lowered to a point that causes steady ebullition without danger of loss. Steam should issue continually for five, ten, or as many minutes as may be deemed necessary or allowable. Then the crucible, still covered, is transferred with the tongs shown in fig. 1 (p. 28) to the titration dish and the operation is concluded as in  $\alpha$ . The method should first be tested by the novice with ferrous-sulphate solution that has been standardized without hydrofluoric acid. It is important not to prolong the boiling unduly, for salts not easily soluble will separate, and as the temperature rises the oxidizing action of the concentrating sulphuric acid may come into play.

The directions given by Pratt<sup>a</sup> with reference to the treatment of a possible undissolved residue of very refractory minerals must be understood as applying only to homogeneous minerals and not to rocks, where the relations of ferrous and ferric iron in the undecomposed portion are certainly different from those in the part dissolved.

$\gamma$ . According to Treadwell.—The specially ground powder in a dish or capacious crucible is placed, after stirring up with 5 or 10 cm.<sup>3</sup> of dilute sulphuric acid, on a support of glass or platinum  $a$  in the lead box C, which in turn is supported in a paraffin bath B, fig. 21. After covering the box with its leaden cover there is introduced through A a rapid current of carbon dioxide<sup>b</sup> until the air is displaced. The cover is then quickly raised. Concentrated hydrofluoric acid (5 to 7 cm.<sup>3</sup>) is added to the dish and the cover is replaced without interrupting the current. The contents of the dish are stirred with a platinum rod through the opening directly over the dish. The bath has meanwhile been brought to about 100°, a temperature which is maintained with occasional stirring for from one-half to one hour or more, according to the resisting quality of the powder. Then the temperature is raised to about 120° for an hour to expel most of the hydrofluoric acid. The flame is then extinguished and the solution allowed to cool as quickly as may be, still in the gas current. When

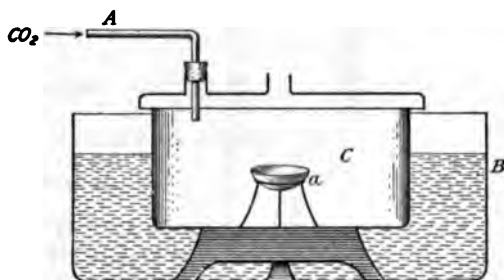


FIG. 21.—Treadwell's apparatus for ferrous-iron determination. B, Paraffin bath; C, lead box with closely fitting cover of same material;  $\alpha$ , platinum dish resting on glass support.

<sup>a</sup>Am. Jour. Sci., 3d ser., vol. 48, 1894, p. 150.

<sup>b</sup>See footnote b, p. 91.

cool, the contents of the dish are titrated, as in  $\alpha$ , in presence of an additional few cubic centimeters of sulphuric acid.

Although the chief object of this method is to remove the excess of hydrofluoric acid and thus avoid one source of error in titration, it may be said, so far as my own limited experience with it goes, that the expulsion is by no means perfect at  $120^{\circ}$  and that the long duration of the experiment renders it likely that lower results will be obtained than with the variations of the hydrofluoric-acid method described in  $\alpha$  and  $\beta$ , by reason of greater oxidation of ferrous iron.

#### D. UNCERTAINTIES OF THE FERROUS-IRON DETERMINATION.

From the foregoing it is apparent that, despite the utmost care in practical manipulation, the exact determination of ferrous iron in rocks is one fraught with extraordinary difficulties and uncertainties. Only in absence of decomposable sulphides and carbonaceous matter and when the amount and condition of vanadium are known can the result be regarded as above suspicion.

### 20. ALKALIES.

#### A. THE J. LAWRENCE SMITH METHOD.

##### a. ITS ADVANTAGES.

The various methods for getting at the alkalies in insoluble silicates differ more in the mode of attack of the mineral powder and in the immediately subsequent treatment than in the final stages. With very few exceptions, since the early days of the Survey's existence, all alkali determinations have been made by the method of J. Lawrence Smith,<sup>a</sup> which is far more convenient than and fully as accurate as those in which decomposition is effected by hydrofluoric and sulphuric acids, or by bismuth, lead, or boric oxides. One of its chief advantages is the entire elimination of magnesia at the start.

##### b. REAGENTS AND APPARATUS.

Decomposition of the powder is effected by heating it with its own weight of ammonium chloride and eight times as much precipitated calcium carbonate.

The ammonium chloride used must be purified, preferably by sublimation, or made by neutralizing pure ammonia by pure hydrochloric acid, and the calcium carbonate is best obtained from pure calcite by solution and reprecipitation. However obtained, this last is rarely free from alkalies, which must be estimated once for all in a blank test in order to apply a correction. Eight grams of the carbonate will yield usually from 0.0012 to 0.0016 gram of alkali chlorides, almost

<sup>a</sup> Am. Jour. Sci., 2d ser., vol. 50, 1871, p. 269; Am. Chemist, vol. 1, 1871; Annalen Chem. und Pharm., vol. 139, 1871, p. 82.

entirely the sodium salt, but the amount has been brought down to half the above by very long washing. This correction may be admitted at once to be a defect of the method, but it is one easily applied with safety. It is not at all certain that all the alkali found in the blank test comes from the carbonate. Much hot water is used for the analysis and a hot-water bottle, even of Jena glass, will afford weighable amounts of alkali.

The ignition may be made in a covered crucible of ordinary shape and of about 20 to 30 cm.<sup>3</sup> capacity, heated to dull redness for not more than two-fifths of its height, but the heat has to be kept so low in this case to avoid loss by volatilization that perfect decomposition is not always assured.<sup>a</sup> Hence, to avoid waste of time in very fine grinding, the form of crucible with cap originally advocated by Smith is very much to be preferred, since it permits, when set at an angle through an opening in the side of a fire-clay cylinder, of the application of the full heat of two burners, and perfect decomposition invariably results without the need of extraordinary care in grinding. The crucible used in this laboratory (fig. 22) for one-half gram of rock powder and 4 grams of calcium carbonate is 8 cm. long, 1.8 cm. wide at the mouth, and 1.5 cm. at the bottom. For double the amounts or more the dimensions are 8 cm., 2.5 cm., and 2.2 cm. The weights are 25 and 40 grams.

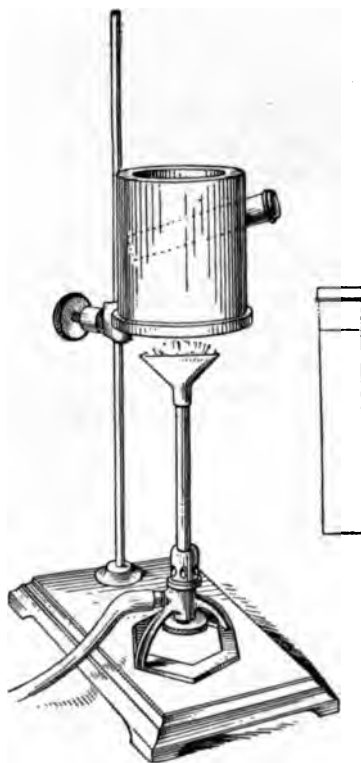


FIG. 22.—The J. Lawrence Smith crucible for alkali determinations. For dimensions see text.

#### C. TREATMENT OF THE MINERAL POWDER.

Perfectly satisfactory results are to be obtained with but half a gram of rock powder. This is weighed out, ground down somewhat finer in a large agate mortar, mixed with its own weight of sublimed ammonium chloride, and the two thoroughly ground together. Then nearly all of 4 grams of calcium carbonate is added and the grinding continued till a thorough mixing has resulted. The contents of the mortar are trans-

<sup>a</sup> R. L. Steinlen (Chem. Zeit., vol. 29, 1905, p. 264) describes a simple cooler for the lid of an ordinary crucible to obviate this difficulty. E. W. Morley (private communication) uses with good effect as a lid a closely fitting small platinum dish filled with cold water.

ferred to the long crucible, the rest of the carbonate being used for rinsing off mortar and pestle. The crucible is then capped and placed in an inclined position in a clay cylinder (fig. 22) or through a hole in a piece of stout asbestos board clamped in a vertical position, and heated for about ten minutes by a low, flat flame placed at considerable distance beneath. As soon as the odor of ammonia is no longer perceptible the nearly full flame of two Bunsen burners is substituted, and continued for forty to fifty minutes. The sintered cake<sup>a</sup> detaches readily from the crucible as a rule; if not, it is softened up in a few minutes by hot water and digested in a dish until thoroughly disintegrated. It is first washed by decantation, and any lumps are broken up by a pestle, then thrown on the filter and well washed with hot water. The residue should dissolve completely in hydrochloric acid without showing the least trace of unattacked mineral, not even of quartz, though sometimes a few black particles of iron ores will dissolve but slowly.

#### d. SEPARATION OF CALCIUM AND SULPHURIC ACID.

All but a trifling amount of the calcium is separated at boiling heat in a large platinum dish by double precipitation by ammonia and ammonium carbonate. The combined filtrates are evaporated to dryness and the ammonium salts are carefully driven off (B, p. 30). From the aqueous solution of the residue—but a few cubic centimeters in bulk—the rest of the calcium is thrown out by ammonia and ammonium oxalate, the last being more effective than the carbonate. The filtrate, caught in an untared platinum crucible or small dish, is evaporated to dryness and gently ignited; the residue is moistened with hydrochloric acid to decompose any alkali carbonate that may have been formed, again evaporated, ignited, and weighed. On solution in water a few tenths of a milligram of fixed residue is invariably left, which should be collected, ignited, and weighed in the same crucible or dish in order to arrive at the weight of the chlorides.

If the rock contains sulphur this will be in part found with the chlorides as sulphate. Therefore, if the sulphur is at all considerable in amount it must be removed by a drop of barium chloride before the final precipitation of the calcium. The excess of barium is removed by ammonium carbonate and the last of the calcium by ammonium oxalate, as above. If the sulphur is not thus removed there is danger, if not certainty, of the potassium chloroplatinate carrying sodium sulphate. A faint reaction for sulphate can usually be obtained, any way, if the evaporations have been made on a water bath heated by gas.

<sup>a</sup> To avoid the formation of a melted cake with silicates high in iron it is advisable to increase the proportion of calcium carbonate. In order to lessen the chance of adhesion to the bottom W. T. Schaller, this laboratory, finds it well to place a layer of calcium carbonate at the bottom of the crucible before introducing the mixture. Mr. Steiger finds that the slacking of the cake is aided by using but 1 or 2 cm. of water at first.

## e. PRECIPITATION OF POTASSIUM.

To the solution of the chlorides in a small porcelain <sup>a</sup> dish an excess of platinic chloride solution is added. The dilution should be such that when heated on the water bath any precipitate that may form wholly redissolves. Evaporation is then carried on till the residue solidifies on cooling. It is then drenched with absolute alcohol <sup>b</sup> or with that of 80 per cent strength, filtered by decantation through a very small filter and washed by decantation with alcohol of the same strength. The precipitate is not brought onto the filter more than can be avoided. Dish and filter are then dried for a few minutes to remove adhering alcohol, the contents of the former are transferred to a weighed platinum crucible or very small dish, and what still adheres to the porcelain is washed through the filter with hot water into the weighed receptacle. This is now placed on the steam bath and afterwards heated for a short time to 135° in an air bath. It is very important to cover the dish at first in the air bath, for decrepitation with resultant loss sometimes takes place if this is not done. With rocks low in potash drying at 100° suffices fully to expel all water. The factor used for reduction of  $K_2PtCl_6$  to  $2KCl$  is 0.307 and of  $2KCl$  to  $K_2O$ , 0.632. It does not seem necessary with the relatively small amounts of potassium in rocks to make use in place of the first of these of the slightly different empirical factor given in some text-books, a factor based on the analysis of commercial potassium salts. If thought desirable the potassium salts can be tested spectroscopically for caesium and rubidium.

## f. SODIUM AND LITHIUM.

If it is desired to check the sodium, ordinarily determined by difference, this can readily be done by evaporation of the alcoholic filtrate from the potassium chloroplatinate, reduction of the platinum by hydrogen or otherwise as mentioned in the footnote, <sup>c</sup> filtration, evaporation, and weighing of the sodium chloride. A check made in this way should agree with the determination by difference within half a milligram on the weight of the chloride. Lithium may then be tested for by the spectroscope, or in case no direct weighing of the sodium chloride is made, the evaporated alcoholic solution may be examined directly. Lithium is almost invariably

<sup>a</sup> Preferred to platinum because of the possibility, under certain rare and ill-understood conditions, of the formation of an insoluble platinous compound, probably by reaction between the platinum of the dish and that of the salt. (See also Bolm, F., *Zeitschr. anal. Chemie*, vol. 38, 1899, p. 349).

<sup>b</sup> H. Precht (*Zeitschr. anal. Chemie*, vol. 18, 1879, p. 513.) claims that this is to be preferred to 80 per cent alcohol, especially if evaporation has been carried to dehydration of the sodium salt. A. Atterberg disputes this final statement and says that 80 per cent alcohol gives better results.

<sup>c</sup> When haste is not an object, this way of Bunsen's for removing platinum from the chlorides of the alkalies is by far the neatest and most satisfactory. The small flask containing the solution is placed in a water bath and attached to a hydrogen generator. After expelling all air the flask is closed, without breaking connection with the generator, and left to itself, except for occasional light shaking up, till reduction is accomplished. A more expeditious and very satisfactory reduction is effected by evaporating the solution to dryness with metallic mercury, then heating to expulsion of the excess of mercury and of its chloride (Sonstadt, E., *Jour. Chem. Soc.*, vol. 67, 1895, p. 984, who thus reduces potassium chloroplatinate in order to weigh its platinum).



present, but almost never in amount to warrant quantitative estimation. Should it be so, however, the very excellent Gooch method (summarized below) of separation by amyl alcohol is to be followed. In rock analysis there need be no fear of enough lithium falling with the potassium to cause any concern.

If ammonium carbonate alone has been relied on to separate all calcium (see d., p. 146) the few tenths of a milligram of calcium chloride that escaped precipitation should now be separated from the sodium and the proper correction made.

*α. Gooch's method<sup>a</sup> for separating lithium.*—To the concentrated solution of the chlorides amyl alcohol is added and heat is applied, gently at first, to avoid danger of bumping, until the water disappearing from solution and the point of ebullition rising and becoming constant for some minutes at a temperature which is approximately that at which the alcohol boils by itself, the chlorides of sodium and potassium are deposited and lithium chloride is dehydrated and taken into solution. At this stage in the operation the liquid is cooled and a drop or two of strong hydrochloric acid added to reconvert traces of lithium hydrate in the deposit, and the boiling continued until the alcohol is again free from water. If the amount of lithium chloride present is small, it will now be found in solution and the chlorides of sodium and potassium will be in the residue, excepting the traces, for which correction will be made subsequently. If, however, the weight of lithium chloride present exceeds 10 or 20 milligrams, it is advisable at this point, though not absolutely essential to the attainment of fairly correct results, to decant the liquid from the residue, wash the latter a little with anhydrous amyl alcohol, dissolve in a few drops of water, and repeat the separation by boiling again in amyl alcohol. For washing, amyl alcohol, previously dehydrated by boiling, is to be used, and the filtrates are to be measured apart from the washings. In filtering it is best to make use of the perforated crucible and asbestos felt, and apply gentle pressure. The crucible and residue are ready for the balance after drying for a few minutes directly over a flame turned low. The weight of insoluble chlorides actually obtained in this manner is to be corrected by the addition of 0.00041 gram for every 10 cm.<sup>3</sup> of amyl alcohol in the filtrate, exclusive of washings, if the insoluble salt is entirely sodium chloride, 0.00051 gram for every 10 cm.<sup>3</sup> if potassium chloride constitutes the residue, and, if both sodium and potassium chlorides are present, 0.00092 gram; but \* \* \* the entire correction may in any case be kept within very narrow limits if due care be given to the reduction of the volume of residual alcohol before filtration. The filtrate and washings are evaporated to dryness, treated with sulphuric acid, the excess of the latter driven off, and the residue ignited to fusion and weighed. From the weight thus found the subtraction of 0.00050 gram is to be made if sodium chloride constitutes the precipitate, 0.00059 gram if potassium chloride alone is present in the residue, and 0.00109 gram if both these chlorides are present, for every 10 cm.<sup>3</sup> of filtrate, exclusive of washings.

Amyl alcohol is not costly, the manipulations of the process are easy, and the only objectionable feature—the development of the fumes of amyl alcohol—is one which is insignificant when good ventilation is available.

The process has been used for some months frequently and successfully, by others as well as by myself, for the estimation of lithium in waters and minerals.

#### B. SEPARATION OF ALKALIES BY OTHER METHODS.

When, as may happen in rare instances, it is necessary to estimate alkalies in the main portion after elimination of silica, alumina, lime, etc., in one of the usual ways, the question of a suitable method for separating magnesium becomes important.

<sup>a</sup> *Proc. Am. Acad. Arts Sci.*, 1886, p. 177; *Bull. U. S. Geol. Survey* No. 42, 1887, pp. 85-88; *Chem. News*, vol. 55, 1887, pp. 18, 20, 40, 56, 78; *Am. Chem. Jour.*, vol. 9, 1887, p. 33.

## a. THE MERCURIC-OXIDE METHOD.

The old barium-hydroxide method is not to be recommended. The mercuric-oxide method of Zimmermann, whereby the magnesia is precipitated from solution of the chlorides by moist, freshly precipitated, and alkali-free mercuric oxide, can give satisfactory results. The oxide is added in excess to the solution in a platinum crucible and evaporated to dryness. Then the mercuric chloride and most or all of the excess of oxide are expelled by cautious heating. On leaching with water the magnesia remains on the filter. With more than 1 per cent of magnesia the operation must be repeated (Dittrich).

## b. THE AMMONIUM-CARBONATE METHOD.

Lately the once favored method of precipitating the magnesium by neutral ammonium carbonate in concentrated solution has been again recommended.<sup>a</sup> The magnesium solution must be as strongly concentrated as possible, and a great excess of ammonium carbonate solution must be used. A voluminous precipitate forms, which dissolves on vigorous stirring if enough of the precipitant was used. After a time a crystalline precipitate falls—a double carbonate of magnesium and ammonium—which is insoluble in the concentrated solution of ammonium carbonate. Allow to stand for six to twenty-four hours. Wash with the concentrated ammonium carbonate solution. It is no exercise of undue caution to redissolve and reprecipitate, to make sure of getting all alkali in the filtrate, especially potassium.

## c. THE AMYL-ALCOHOL METHOD.

Under certain circumstances, notably absence of lithium, the method of Gooch developed by Riggs<sup>b</sup> may be satisfactory. It is similar to that of Gooch for separating lithium from sodium and potassium chlorides by amyl alcohol, and involves the same solubility corrections for the alkali chlorides above noted (f. *α*, p. 148) in the description of Gooch's method. Riggs's summary is as follows:

Evaporate the solution nearly or quite to dryness. Dissolve the residue in as little water as possible and add a few drops of hydrochloric acid. Then add 30 to 40 cm.<sup>3</sup> of amyl alcohol and expel the water by bringing the alcohol to boiling. Continue the boiling until the volume of the solution is reduced to 10 cm.<sup>3</sup>, or even considerably less. In filtering, it is of great advantage to use a perforated crucible and an asbestos felt and to filter under pressure. In case the total chlorides exceed 0.2 gram it may be advisable to decant the liquid, wash the residue, redissolve, and repeat the precipitation. If this be not done, the precipitate should be redissolved with the least possible quantity of water, a few drops of hydrochloric acid added, and the precipitation repeated in the original solution. The filtrate is transferred to a weighed platinum dish and evaporated. Water is added before the alcohol has been expelled, and the evaporation continued. The residue is dissolved

<sup>a</sup>Wülfing, E. A., Ber. Deutsch. chem. Gesell., vol. 32, 1899, p. 2214. The neutral carbonate is prepared by dissolving 230 grams of ammonium carbonate in 180 cm.<sup>3</sup> of ammonia of 0.92 specific gravity and enough water to make 1 liter. This is sometimes called Schaffgotsch's solution.

<sup>b</sup>Am. Jour. Sci., 3d ser., vol. 41 1892, p. 103.

in water. Sulphuric acid is added in slight excess. This solution is evaporated to dryness, the residue ignited and weighed, and the treatment with sulphuric acid is repeated. The residue of insoluble chlorides may be transferred to the weighed perforated crucible and dried at a temperature below their melting points, or it may be dissolved and the solution transferred to a weighed platinum dish, evaporated, and the residue dried as above and weighed.

As with the Gooch method for lithium, the numerous test results are good.

## 21. CARBON DIOXIDE, CARBON.

### A. QUALITATIVE TEST FOR CARBON DIOXIDE.

In the preliminary qualitative test for carbon dioxide, it must be remembered that while calcite gives off its carbon dioxide on treatment with cold acid, dolomite and siderite do not, and hence warming should not be omitted; otherwise, a few tenths per cent of carbon dioxide can very well be overlooked. Moreover, the powder should first be agitated with a little hot water, to remove all entangled air which might otherwise be mistaken for carbon dioxide. In order not to overlook traces the test is best made in a test tube, first boiling the powder with a little water, then cooling and adding dilute hydrochloric acid. If effervescence is immediate the presence of calcite is assured; if escape of bubbles takes place only on warming the carbonate is not calcite. In order to make sure of the reaction it may be necessary to use a pocket lens, holding the test tube in an inclined position and looking down in order better to see the minute bubbles as they stream up along the upper glass wall. It is, of course, important not to mistake escaping hydrogen sulphide for carbon dioxide.

### B. QUANTITATIVE TESTS FOR CARBON DIOXIDE.

#### a. DIRECT METHOD.

For the quantitative determination of carbon dioxide an apparatus permanently set up is used, of which several forms have been described by different writers.<sup>a</sup> The one depicted in fig. 23 is a compact arrangement at present used in the Survey laboratory.

The rock powder (1 to 5 grams) is boiled with dilute hydrochloric acid in a small Erlenmeyer flask attached to an upward-inclined condenser, whence, after passing through a drying system—calcium chloride, anhydrous copper sulphate to retain hydrogen sulphide from decomposable sulphides and any hydrochloric acid that may pass over—then calcium chloride again—the carbon dioxide is caught by absorption tubes filled with soda lime followed by calcium chloride. Of course arrangement is made for a current of  $\text{CO}_2$ -free air with which

<sup>a</sup> For a simple and convenient form, see Treadwell's Quantitative Analysis, American edition, p. 226

to sweep out the apparatus before and after the experiment, and for a slow current during its continuance. The results are very accurate and the determination can be quickly carried out.

The manipulations are as follows: Hot water is poured upon the powder in the flask; this is attached to the condenser, and a current of  $\text{CO}_2$ -free air is forced through the whole system, except the weighed

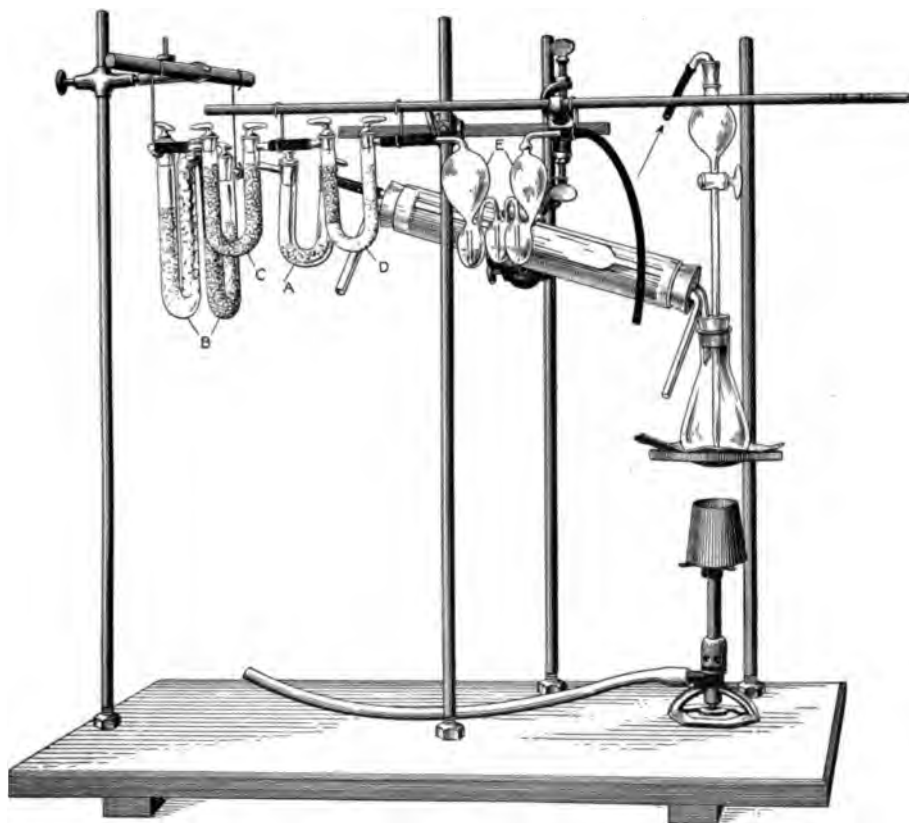


FIG. 23.—Compact form of apparatus for determination of carbon dioxide. A, Protective calcium-chloride tube, frequently refilled, to retain most of the moisture from the condenser. B, Double U-tube, filled in the center with pumice impregnated with anhydrous copper sulphate and at both ends with calcium chloride. C and D, absorption tubes, C and one-third of D containing soda lime, followed in D by calcium chloride. C needs to be refilled before the soda lime in D has absorbed much if any carbon dioxide, D then requires only occasional refilling, but it will not do to use it indefinitely, since the calcium chloride in it becomes less and less effective as it takes up the water set free from the soda lime in C during its conversion to carbonate. E, Observation bulbs containing sulphuric acid to show the rate of gas flow.

absorption tubes, until the original air has been displaced, the observation bulbs E being attached directly to the drying system, B. The stopcock in the separatory funnel is then closed, the latter half filled with hydrochloric acid (1:1), the rubber stopper of the funnel replaced, the absorption tubes inserted between B and E, and the acid allowed to flow into the flask, slowly if there is much carbon dioxide

rapidly if there is but little. When effervescence diminishes in the former case, at once in the latter, the lamp is lighted and the flow of water through the condenser started. The flame is kept low, so as to secure steady but quiet ebullition, and the gas current is not interrupted, although reduced to a slow rate. With much carbon dioxide the rate of absorption is very readily noted by holding the hand to the soda-lime tubes, which become hot or warm where absorption is taking place. A sufficient time having elapsed, the flame is extinguished and the air current increased. When cool the tubes C and D are weighed after remaining some time in the balance case.

The soda lime must be porous, not hard and unabsorptive like that sometimes used for the combustion of nitrogenous organic substances.

For the simultaneous determination of carbonates and the carbon of carbonaceous matter see C., page 152.

It has already been shown under "Water" (5 C. c, p. 66) how in case of need, as paucity of material, the determination of carbon dioxide can be combined with that of water by fusion with lead chromate. Soda-lime absorption tubes then follow the calcium-chloride tube in carrying out the determinations there referred to.

#### b. INDIRECT METHOD.

The older forms of apparatus for the indirect determination of carbon dioxide are too well known to need description. Some are fragile or cumbersome and great care is requisite to the attainment of good results. The form recently devised by J. L. Kreider<sup>a</sup> (fig. 24) has the merit of simplicity, cheapness, and ease of manipulation. It is adapted for determining not only carbon dioxide, but also other volatile substances produced by a variety of reactions, as hydrogen resulting from the action of an acid on certain metals, nitrogen from urea, ammonium oxalate or ammonium chloride by the action of sodium hypobromite, etc. Kreider's test results are excellent, though with carbonates generally low to the extent of about 0.06 to 0.1 per cent.

FIG. 24.—Kreider's apparatus for indirect determination of volatile substances.

The apparatus \* \* \* is light and easily made from three test tubes. \* \* \* The test tube A is changed in no way from its original form; B is perforated in the bottom with a hole about 1 cm. in diameter and fits tightly within A; and C, so selected that it fits loosely within B, is drawn out to a small capillary tube.

When the apparatus is to be used, the capillary of C is pushed through the hole of B, packed loosely with cotton; B is filled to the depth of from 6 to 8 cm. (about two-thirds of its contents) with granular calcium chloride, and B and C are adjusted as shown.

To the test tube, C, is fitted a one-holed stopper, through which passes a short glass tube

<sup>a</sup> *Am. Jour. Sci.*, 4th ser., vol. 19, 1905, p. 188; *Zeitschr. anorg. Chemie*, vol. 44, 1905, p. 154.

which is closed by a rubber cap and plug. Upon removing the plug and applying suction to the short tube the reagent employed to liberate the volatile product to be determined is drawn up through this capillary until C is sufficiently filled. Upon replacing the plug the reagent remains within C, held by atmospheric pressure.

The tubes A and B may be so selected that very little of the product evolved can escape between them, but in case they fit very loosely a ring of paraffin melted into the mouth of A, about B, by means of a hot iron or wire, seals the joint securely. A very convenient way to attach the paraffin is to melt it between A and another tube, which fits A as does B and may be removed by a turning motion, leaving the ring into which B will fit, and which then requires very little heating to make a tight joint. If care be used in taking apart A and B at the close of an experiment, such a ring of paraffin remains in place and may be used many times without replacement, being remelted by a touch of the hot wire before every new experiment.

In making the determination the substance under examination is weighed and placed in the bottom of A. The reagent to be employed, 10 to 15 cm.<sup>3</sup>, is drawn into C and held there in the manner described. The test tube A is slipped over B and this joint is sealed with paraffin, as has been shown. The apparatus is wiped, placed on the balance, and weighed.

Upon removing the cap from the small tube in C the reagent runs from C into A. The volatile product is formed, is forced upward through the drying column of calcium chloride, and escapes through the annular space between B and C. When the action ceases a current of dry air is forced through C to drive all the volatile products from the apparatus, the cap is then replaced, and the whole placed on the balance to be weighed. The loss in weight represents the volatile product.

#### C. CARBON.

Fusion with lead chromate is commonly resorted to in order to determine the carbon of graphite or carbonaceous matter in rocks and ores, though boiling in presence of sulphuric and chromic acids can be made to yield good results.

If carbonates are at the same time present they can be determined as in B (p. 150), and by a second determination, as just referred to, the total carbon in both forms can be found as CO<sub>2</sub>, whence that of the graphite is derived by difference. Or, according to G. T. Morgan,<sup>a</sup> the carbonates are first decomposed as in B by orthophosphoric instead of hydrochloric acid, whereupon chromic acid is added and the carbon oxidized and determined as CO<sub>2</sub>.

## 22. CHLORINE.

#### A. CONDITION IN ROCKS.

Chlorine may exist in rocks in a water-soluble condition, in minerals that are decomposed by nitric acid, and in those not attacked by this acid. In the first form it doubtless exists as infiltrated sodium chloride or as original inclusions in one or more of the constituent minerals; in the second in minerals of the sodalite group and sometimes apatite; in the last chiefly in the scapolites. Where

<sup>a</sup> Jour. Chem. Soc., vol. 85, 1904, p. 1001.

a qualitative test shows the water-soluble form to be present in determinable amount, it must be separately extracted and the amount found deducted from the total as determined in a second portion. Sometimes it may be possible to use the extracted material for the determination of the rest of the chlorine.

#### B. DETERMINATION OF WATER-SOLUBLE CHLORINE.

A suitable amount, which may be several grams, of the powder is extracted with water. The filtrate is very often turbid, a condition which may sometimes be prevented by using double filters or by adding some chlorine-free salt, like sodium nitrate, to the wash water. If the filtrate remains persistently cloudy the precipitation of silver chloride may be proceeded with as usual, after acidifying with nitric acid. It is best to let the precipitate settle over night in order to obtain a clear filtrate on the morrow. It is collected on a small filter, washed with water acidified with nitric acid, dried, and ignited in a very small porcelain crucible without allowing the paper to burst into flame. Most of the chloride will be reduced to metal. On disappearance of all carbon a drop of nitric acid is added and evaporated, then similarly a drop of hydrochloric acid, and the crucible gently heated, but not to fusion of the silver chloride, and weighed. The chloride is then dissolved in a few drops of warm ammonia water and separated by filtration from the siliceous matter, which latter is ignited and weighed in order to get the weight of the chloride.

This method of procedure is permissible even when the original aqueous extract was quite clear, though the operation then stops with the weighing of the chloride. Or, if very small in quantity, the dry paper with its contents is wound up in a tared platinum wire and carefully ignited after Bunsen's manner. The increased weight of the wire is due to the metallic silver of the chloride which has alloyed with it.

#### C. DETERMINATION OF ACID-SOLUBLE CHLORINE.

##### a. BY NITRIC ACID.

If nitric acid will decompose the chlorine-bearing minerals, or if it is desired to distinguish between the chlorine in soluble and in insoluble minerals, the rock powder may be boiled for a few minutes with dilute chlorine-free nitric acid. The acid should be very dilute in order to cause no loss of chlorine and the boiling should be as brief as possible for the same reason, and also to prevent gelatinization of the silica from soluble silicates. Acid as dilute as 1:40 will readily decompose apatite and probably the minerals of the sodalite group, but it is doubtful if anything like this dilution is called for.

The filtrate does not require evaporation for the removal of dissolved silica, but may be precipitated at once with silver nitrate. The precipitate is treated as in B, or if at all appreciable in amount may be collected on a Gooch crucible.

**b. BY NITRIC AND HYDROFLUORIC ACIDS.**

In many cases in order to obtain all the chlorine it is sufficient to attack the powder by chlorine-free hydrofluoric and nitric acids in the cold, with occasional stirring, and after filtering through paper fitted into a rubber funnel or large platinum cone to throw down the chlorine by silver nitrate. The presence of nitric acid is necessary, since in its absence ferrous fluoride reduces silver nitrate with deposition of crystallized silver. It may be advisable to redissolve the chloride on the filter in ammonia and to reprecipitate by nitric acid and a drop of silver nitrate. It seems difficult to obtain in the market hydrofluoric acid free from a trace of hydrochloric.

**D. DETERMINATION OF CHLORINE BY ALKALI FUSION.**

Chiefly for the last reason, in order to make sure of getting all the chlorine, it is best to fuse with chlorine-free sodium-potassium carbonate, or even sodium carbonate alone, first over the full burner, then for a moment or two over the blast, to leach with water, acidify in the cold with nitric acid, and precipitate by silver nitrate without preliminary separation of silica. After acidification in the cold, except in a very concentrated solution, no precipitation of silica need be feared. The treatment of the precipitate will be as in B and C, according to its amount and freedom from contamination.

**23. FLUORINE (SILICA IN PRESENCE OF FLUORINE.)**

**A. IMPERFECTION OF THE QUALITATIVE TEST FOR FLUORINE.**

There is no qualitative test which will reveal with certainty the presence of fluorine in rocks. Heating the powder before the blow-pipe with sodium metaphosphate on a piece of curved platinum foil inserted into one end of a glass tube, or in a bulb tube, is not to be relied on in all cases. While as little as 0.1 per cent of fluorine can sometimes be thus detected with ease, much larger amounts in another class of rocks may fail to show.

**B. QUANTITATIVE DETERMINATION OF FLUORINE.**

**a. THE METHOD.**

For the reason that the fluorine minerals occurring in rocks are mostly attacked but partially, if at all, by strong hot sulphuric acid, none of the methods depending on the volatilization of silicon fluoride can be used. There is the further reason that even with decomposable



fluorides it is impossible to obtain quantitative results in presence of amorphous silica or silicates.<sup>a</sup> Only with quartz powder is volatilization perfect. The method to follow is that of Berzelius, which, though not difficult, requires great attention to details and even then affords low results (see c for causes of error). It admits of the determination of silica at the same time, and is in fact the only method available for silica in fluorides.

Two grams of the rock powder are fused with four to five parts of sodium-potassium carbonate free from fluorine, avoiding the use of a blast if possible. For minerals rich in fluorine and low in silica it may be necessary to add pure silica before fusing in order to effect complete decomposition of the fluoride, just as with the alkaline-earth phosphates. But this will probably never be required in ordinary rock analysis. To the aqueous extract, containing the fluorine and usually much of the silica besides other bodies, several grams of ammonium carbonate are added, the liquid is digested at a gentle heat (40°) for some time, and on cooling more carbonate is introduced.<sup>b</sup> After twelve hours the precipitate is collected and washed with water containing ammonium carbonate. From the filtrate the excess of the latter is expelled by evaporating nearly to dryness and the somewhat diluted solution is brought toward neutrality as follows, according to the directions of Treadwell. A few drops of phenolphthalein are added, then nitric acid (not hydrochloric in this case, see below) till the red color disappears. The solution is now boiled, the color reappearing, to be again discharged by acid on cooling. These operations are repeated till it requires but 1 to 1½ cm.<sup>3</sup> of  $\frac{2}{N}$  acid to discharge the color. There is now added 1 to 2 cm.<sup>3</sup> of an ammoniacal solution of zinc oxide,<sup>c</sup> and the liquid is boiled till the ammonia is wholly expelled. The precipitate, containing the last of the silica and some phosphorus, in addition to that thrown down with alumina by ammonium carbonate, is separated and washed with water.

The above-prescribed use of nitric instead of hydrochloric acid for neutralizing is necessitated by the fact that phosphorus, which is

<sup>a</sup> See Daniel, K., *Zeitschr. anorg. Chemie*, vol. 38, 1904, pp. 257-306.

<sup>b</sup> The use of ammonium nitrate or chloride, instead of carbonate, for throwing out the silica and alumina is not to be recommended because of loss of fluorine on subsequent evaporation (H. Rose).

<sup>c</sup> Made by precipitating pure zinc-chloride solution with potassium hydroxide, filtering, washing and dissolving the precipitate in ammonia. If the rocks are very basic, it may happen that the amount of silica in the alkaline solution of the fusion is so small that ammonium carbonate may be dispensed with and the zinc-oxide solution added at once, after neutralizing as above.

Instead of the ammoniacal zinc-oxide solution F. Seemann (*Zeitschr. anal. Chemie*, vol. 44 1905, p. 343) advocates using the Schafigotsch solution (p. 119, footnote) in which has been dissolved in the cold 20 grams of freshly precipitated mercuric oxide to the liter. For every 0.2 gram of dissolved silica 100 cm.<sup>3</sup> of this reagent are to be used. The solution is then to be evaporated to complete dryness, the residue taken up with water, filtered and washed, the filtrate again neutralized with hydrochloric acid [nitric in rock analysis—W. F. H.], and again evaporated to remove the last of the silica. Where silica is to be determined at this stage this reagent has the advantage over the zinc solution that direct ignition of the precipitates (under a good draft hood) yields at once silica. Its use is manifestly allowable in rock analysis only after the alumina has first been separated by ammonium carbonate.

almost invariably present, and chromium must still be removed, and this can be done only from a nitric solution, as follows: To the still alkaline solution silver nitrate is added in excess, whereby phosphate, chromate, chloride if chlorine is present, and carbonate of silver are precipitated. The last serves to correct any acidity resulting from the reaction between the alkaline phosphate and the silver salt, thus producing the neutral solution needed for complete precipitation of the phosphate and chromate. After slight heating and filtering, the

excess of silver is removed by sodium chloride and 1 cm.<sup>3</sup> of  $\frac{2}{N}$  sodium-carbonate solution is added to the filtrate, which is then boiled in a dish of good size with a large excess of calcium-chloride solution. At this stage there must be no ammoniacal salts in solution, otherwise calcium fluoride may be held up. The precipitate consists of a mixture of calcium carbonate and fluoride, the former serving to aid filtration of the latter. The precipitate is collected on paper and washed with hot water, dried, and ignited separately from the filter as far as possible. If the dish used for the precipitation was of platinum it is not necessary to cleanse it from adhering precipitate, but the contents of the filter are washed back into the dish, the liquid in this is evaporated away, the ash of the filter added, and the dish heated over a free flame to faint redness for a few moments.

Dilute acetic acid is now poured upon the thus gently ignited mixture in small quantities at a time and in amount slightly in excess of that needed to dissolve the calcium carbonate. When visible action ceases the liquid is evaporated to dryness and the residue taken up with hot water and a drop of acetic acid, filtered, dried, and gently ignited.

The amount of fluoride obtained from rocks being usually very small, a single treatment as above suffices, but for the larger amounts obtained from fluorine minerals a second and even third treatment with acetic acid may be needed, filtering and igniting after each. S. L. Penfield and J. C. Minor<sup>a</sup> found in such cases that if a great excess of acetic acid is used at the start the results are lower than by the repeated treatment. For considerable amounts, when the loss between two treatments is reduced to not more than half a milligram, F. P. Treadwell and A. A. Koch<sup>b</sup> take the next to the last weight as correct.

#### b. TESTING OF THE CALCIUM FLUORIDE.

The well-washed and gently ignited calcium fluoride finally obtained in the course of this method should be converted to sulphate as a check on its purity, and at the same time as a qualitative test to ascertain if it really is calcium fluoride by the characteristic odor of the gas given off. Should fluorine be found, and the weight of sulphate not

<sup>a</sup> *Am. Jour. Sci.*, 3d ser., vol. 47, 1894, p. 389.    <sup>b</sup> *Zeitschr. anal. Chemie*, vol. 43, 1904, p. 469.

correspond to that of the fluoride, the former should be dissolved in hot nitric acid and tested for phosphorus by ammonium molybdate solution. If phosphate is absent the impurity may have been silica or calcium silicate—which of these it would be difficult to decide. In the former case the fluorine might be safely deduced from that of the sulphate, but not in the latter. If the rock were rich in sulphur it might happen that calcium sulphate would be thrown down with the fluoride, but this should be removed by thorough washing. If not, and it were certainly the only impurity present, the fluorine could be calculated, after conversion of the fluoride into sulphate, by the formula.

$\text{CaSO}_4 - \text{CaF}_2 : 2 \text{ F} :: \text{Diff. between impure CaSO}_4 \text{ and CaF}_2 : x.$

It is an exceptional case when there is exact agreement between the weight of fluoride and sulphate, and with the small amounts usually met in rocks the error may be an appreciable one in percentage of fluorine, though of no great significance otherwise.

#### C. ACCURACY OF THE METHOD.

It is evident from the above that if the greatest care is not taken to prevent any kind of contamination of the weighed fluoride the error in the small amounts in question may be very great. If phosphate were present it would not do to apply the check by conversion to sulphate, because the evaporation with sulphuric acid and following ignition would volatilize metaphosphoric acid.

There is another source of error due to the solubility of the fluoride itself in water and acetic acid. F. P. Treadwell and A. A. Koch<sup>a</sup> have investigated the solvent effect of these and some other reagents. They find it possible to detect additional fluorine by repeating the precipitation with calcium chloride in presence of sodium carbonate, and the total errors to be such that for 5 milligrams  $\text{CaF}_2$  or less the results are much too low, though the qualitative detection can be still made in 100 cm.<sup>3</sup> of solution with only 0.0009 gram  $\text{CaF}_2$ , notwithstanding the fact that the quantitative tests had seemed to indicate that the loss would be about 0.0015 gram  $\text{CaF}_2$  for every 100 cm.<sup>3</sup>, including wash water. It thus appears that with 1 gram of sample amounts of less than 0.04 to 0.05 per cent of fluorine will entirely escape observation.

A source of error not generally known arises whenever calcium fluoride is ignited in contact with paper. Fluorine escapes to a slight extent and is replaced by oxygen this reaction being doubtless due to the action of water vapor and not to oxygen of the air.

F. Seeman<sup>b</sup> has subjected all the known methods of determining fluorine to extended examination, using calcium fluoride as his test material, and declares the Berzelius method to be far inferior to those based on volatilization of the fluorine as silicon fluoride, by reason of

<sup>a</sup> *Zeitschr. anal. Chemie*, vol. 43, 1904, p. 469.

<sup>b</sup> *Zeitschr. anal. Chemie*, vol. 44, 1905, p. 343.

great and varying losses, the causes of which he was unable to satisfactorily trace. He was unable to recover by it more than about 87 to 89 per cent of the fluorine. My tests do not confirm his very unfavorable results. I was able repeatedly to regain 95 to 98 per cent of the fluorine. To do so requires, however, refusion of the residue left after leaching the alkali-carbonate melt, including in the same refusion the silica precipitated by ammonium carbonate and ammoniacal zinc oxide, the treatment of the filtrate from the precipitate of calcium carbonate and calcium fluoride with additional sodium carbonate and calcium chloride, and the reevaporation of the acetic-acid solutions of the precipitated calcium carbonate.

#### C. DETERMINATION OF SILICA.

The several precipitates obtained by ammonium carbonate and zinc oxide, together with the residue from the original alkali fusion, are dissolved as far as may be in hydrochloric acid, the papers ignited and their ashes added, and silica separated by the processes detailed in 6, page 68. These may be greatly simplified, if bases are not likewise to be determined, by evaporating the first filtrate from the silica with much sulphuric acid as in the last part of f.  $\alpha$ , page 88.

### 24. SULPHUR.

#### A. DETERMINATION OF ITS CONDITION.

Before proceeding to the determination of sulphur, its condition, if present, should be ascertained.

Evolution of hydrogen sulphide on boiling with diluted hydrochloric acid is evidence of a soluble sulphide, usually pyrrhotite, but possibly lazurite. Extraction of magnetic particles reacting for sulphur shows pyrrhotite to have been in part at least the source of the hydrogen sulphide. A reaction for sulphuric acid in the filtered solution indicates a soluble sulphate, usually a silicate sulphate, noselite, or hauynite. If the residue, when well washed and treated with aqua regia or hydrochloric acid and bromine, gives more sulphuric acid, the probable presence of pyrite is shown. Should this solution likewise show arsenic, the sulphide may be arsenopyrite, which, however, is of very rare occurrence in igneous rocks, if, indeed, it is ever found there.

It is most uncommon to find any sulphur in rocks in the ordinary simple sulphate condition, except sometimes water-soluble traces, even when considerable pyrite is in evidence. The barium of fresh and even moderately decomposed rocks is apparently always in silicates and not in combination with sulphur.

## B. QUANTITATIVE DETERMINATION.

## a. TOTAL SULPHUR.

Although the sulphur of sulphides may sometimes be correctly determined by extraction with aqua regia or some other powerful oxidizer, in the wet way, this is not always so. Therefore it is better by far to fuse with sulphur-free sodium carbonate and a little niter over the Bunsen burner, and for a few moments over the blast, using throughout the heating the arrangement shown in fig. 3 (p. 30), in order to exclude access of sulphur from the flame gases. This determination may very conveniently be combined with those of barium, titanium, zirconium, and rare earths (p. 116). After thorough disintegration of the fusion in water, to which a drop or two of alcohol has been added for the purpose of reducing and precipitating any manganese dissolved as manganate, the solution is filtered and the residue washed with a dilute solution of sodium carbonate. In the filtrate (100 to 250 cm.<sup>3</sup> in bulk) the sulphur is precipitated at boiling heat or on the steam bath by barium chloride in excess, after slightly acidifying in the cold by hydrochloric acid. Evaporation to dryness first with acid, in order to eliminate silica, is needless, for in the above bulk of solution there will almost never be the least separation of silica with the barium sulphate.<sup>a</sup> It is well that this is so, for evaporation on a bath heated by gas to remove silica would in many cases involve an error fully equal to the sulphur present by contamination from the sulphur of the gas burned. With a steam bath this difficulty does not present itself.

Owing to the small amount of sulphur in rocks, special purification of the barium sulphate obtained is hardly ever needful, especially as it has been precipitated in the absence of iron. Should there be fear of a trace of silica being present, it can be removed by a drop of hydrofluoric and sulphuric acids before weighing the barium sulphate.<sup>b</sup>

## b. FRACTIONAL DETERMINATIONS.

If traces of water-soluble sulphates are present, they can be removed when desired by boiling water.

For the quantitative extraction of the sulphur of all soluble sulphates, simple boiling with hydrochloric acid suffices, which should be done in an atmosphere of carbonic acid if pyrites or other oxidizable sulphides are present, and should be finished as quickly as possible in order to minimize the error resulting from oxidation to sulphuric acid

<sup>a</sup> The results of C. W. Stoddart (Jour. Am. Chem. Soc., vol. 24, 1902, p. 852) show, in the determination of sulphur in coals by the Eschka method, often higher sulphur when silica is not removed by evaporation in the usual way, but this is contrary to all my experience in silicate work, where duplicates agree usually exactly, and if they differ are as likely to be high by one way as by the other. The explanation may possibly be that Stoddart acidified his solutions hot or used too strong an acid and had some silica precipitated.

<sup>b</sup> In Jour. Soc. Chem. Ind., vol. 24, 1905, p. 10, and Chem. Zeit., vol. 29, 1905, p. 29, I. Pattinson and J. T. Dunn call attention to certain sources of error in the determination of the sulphate ion as the presence of sulphides in some barium chloride and the use of red rubber stoppers.

of the sulphur of sulphides, if present, by any ferric salts that may have been dissolved.

If soluble sulphates and sulphides as well as insoluble sulphates and sulphides are present together, the sulphur of the first is found in solution after extraction by hydrochloric acid in a carbon-dioxide atmosphere, and that of the decomposable sulphides by collecting the hydrogen sulphide evolved.<sup>a</sup> In the residue the sulphur of the insoluble sulphides can be estimated, or from the total sulphur found in another portion its amount can be calculated. The error involved in the above estimation of the sulphur of soluble sulphides, due to the possible reducing effect of hydrogen sulphide on ferric salts, is probably negligible. Most of the hydrogen sulphide would be expelled before any such action could take place and probably before the ferric salts were largely attacked, but of course the small proportion of sulphur set free as such from pyrrhotite would escape estimation and introduce further uncertainty. In general, it would be safe enough to assume the composition  $\text{Fe}_7\text{S}_8$  for pyrrhotite. However carefully all these separate determinations may be carried out, the final figures for ferrous and ferric oxides can hardly be regarded as more than approximations when much sulphide is present (see p. 138, c.  $\alpha$ ).

## 25. BORON.

To the best of my belief it has never been found necessary in this laboratory to determine boron quantitatively in a silicate rock. Should this determination be required it would be necessary, since most borosilicates are insoluble minerals, to fuse with sodium carbonate and apply in one of its modifications the Rosenbladt-Gooch method,<sup>b</sup> depending on expelling the boron as methyl ester by distil-

<sup>a</sup> With pyrrhotite a small fraction of its sulphur—one-eighth if the formula  $\text{Fe}_7\text{S}_8$  is adopted—is liberated as free sulphur and not as hydrogen sulphide.

<sup>b</sup> Th. Rosenbladt (*Zeitschr. anal. Chemie*, vol. 26, 1887, p. 21) used magnesia for binding the boron, while F. A. Gooch (*Proc. Am. Acad. Arts Sci.*, 1886, p. 167; *Bull. U. S. Geol. Survey* No. 42, 1887, p. 64; *Chem. News*, vol. 55, 1887, p. 7) preferred lime, as more active and reliable. Gooch and L. C. Jones have later (*Am. Jour. Sci.*, 4th ser., vol. 7, 1899, p. 34, *Chem. News*, vol. 79, 1899, pp. 99, 111) upheld the use of lime and proposed as a convenient though perhaps not quite so perfect substitute, sodium tungstate containing an excess of tungstic oxide. In this article they likewise indicate the precautions now used to insure complete collection and retention of the boron.

For a useful modification in the way of collecting the boric ether in ammonia before bringing in contact with the lime, see S. L. Penfield and E. S. Sperry (*Am. Jour. Sci.*, 3d ser., vol. 34, 1887, p. 222); also H. Moissan (*Comptes rendus*, vol. 116, 1893, p. 1087; *Bull. Soc. chim.*, vol. 12, 1894, p. 955), who modifies the Gooch distilling apparatus in certain respects.

G. W. Sargent (*Jour. Am. Chem. Soc.*, vol. 21, 1899, p. 858) obtained satisfactory results with tourmaline by collecting the boron in sodium-hydroxide solution, neutralizing the alkali with hydrochloric acid by the aid of methyl orange, then titrating the freed boric acid by sodium hydroxide in presence of glycerin, using phenolphthalein as indicator (method of Thompson, *Jour. Soc. Chem. Ind.*, vol. 12, 1893, p. 432).

The latest contributions to the application of this method are by R. J. Manning and W. R. Lang (*Jour. Soc. Chem. Ind.*, vol. 25, 1906, p. 396), who determine the boron in the distillate gravimetrically as the barium salt  $\text{Ba}(\text{BO}_2)_2$ , and by Wilson H. Low (*Jour. Am. Chem. Soc.*, vol. 28, 1906, p. 807) who modifies the distillation feature, with much apparent advantage, by the addition of dry granulated calcium chloride, and perfects the turmeric-paper qualitative test.

lation with methyl alcohol from an acid solution and collecting this in some suitable manner. For simple borates, natural or artificial, this method gives entire satisfaction when all needful precautions are carefully observed, but its application to borosilicates yet needs investigation, in view of the as yet unexplained very discordant results obtained some years ago by J. E. Whitfield in this laboratory on the mineral warwickite, a borotitanate of magnesium and iron. Perhaps his difficulty arose from the fact that a single fusion with an alkali carbonate does not remove all the boron from a borosilicate, as shown by G. W. Sargent<sup>a</sup> and W. E. Ford,<sup>b</sup> a cause which would doubtless be inoperative in the case of rocks, by reason of the small amounts of boron involved. For this last reason it is quite possible also that the accurate determination of but a few milligrams at most of boric oxide would not be feasible by any one of the proposed modifications of collecting and weighing or titrating it. In any case fluorine, if present, would have to be first removed by calcium nitrate or acetate before freeing the boron.

#### 26. CERTAIN CONSTITUENTS IN MINUTE TRACES.

If, as sometimes may happen, the problem is presented of examining rocks for traces of gold, silver, and other elements which are not ordinarily looked for, as in F. Sandberger's investigations bearing on the origin of the metalliferous contents of veins, large weights of material must be taken, up to 50 grams or more. This involves the use, also, of large quantities of reagents, the purity of which must then be looked to with the utmost care. Special directions to meet such cases can not now be given, nor even a complete reference list of the scanty and scattered literature on this subject. Sandberger's own writings deal but little with its analytical side, and from its inaccessibility in the Washington libraries I am as yet unacquainted with the report by H. Von Foullon<sup>c</sup> "Ueber den Gang und die Ausführung der chemischen Untersuchung," following Sandberger's own paper<sup>d</sup> in the general report "Untersuchungen der Nebengesteine der Pribramer Gänge." I have published a few data as to gold, silver, lead, zinc, etc.,<sup>e</sup> in S. F. Emmons's report on "The Geology and Mining Industry of Leadville;" J. S. Curtis,<sup>f</sup> in his report on "The Silver-Lead

<sup>a</sup> Jour. Am. Chem. Soc., vol. 21, 1899, p. 858.

<sup>b</sup> Am. Jour. Sci., 4th ser., vol. 14, 1903, p. 195.

<sup>c</sup> Jahrbuch der Bergakademie, Leoben u. Pribram, 1887, p. 363.

<sup>d</sup> From Sandberger's report it appears that the rocks were treated successively with water, acetic acid, boiling dilute hydrochloric acid for two days, and finally hydrofluoric acid, the several extracts and final residue of fluorides (and pyrite) being separately examined for heavy metals. The products of distillation were also examined. A striking fact observed in all cases was the complete insolubility of the pyrite, even after the severe treatment mentioned. This speaks strongly in favor of the correctness of ferrous iron estimations in silicates by the hydrofluoric and sulphuric acid method when pyrite is present unaccompanied by other sulphides. (See a, p. 138.)

<sup>e</sup> Mon. U. S. Geol. Survey, vol. 12, 1886, Appendix B, pp. 592-596.

<sup>f</sup> Mon. U. S. Geol. Survey, vol. 7, 1884, pp. 120-138.

Deposits of Eureka, Nev.," has given his method of assaying rocks for traces of gold and silver; in volume 7 of the Reports of the Missouri Geological Survey, page 740, are to be found the methods used by James D. Robertson for the determination of lead, zinc, and copper in silicate and carbonate rocks of Missouri. The earlier work of J. G. Forchhammer<sup>a</sup> and L. Dieulafait,<sup>b</sup> the latter of whom examined many hundreds of Archean rocks, was of a qualitative nature.

## 27. THE GASES AND VAPORS EXPELLED BY HEAT.

That rocks and minerals evolve large quantities of gases and vapors when heated is a well-recognized fact. Numerous analyses have shown that the volatile products usually comprise hydrogen and carbon dioxide as preponderating constituents, with carbon monoxide, methane, nitrogen, hydrogen sulphide, etc., in smaller amounts. The total volume may be many times that of the solid. To a certain extent, sometimes a large extent, especially as to carbon dioxide, the source of these is entrapped fluid inclusions, whose origin was contemporaneous with that of the containing minerals. A further portion may have been held dissolved in the rock, particularly in those not wholly crystallized. But from the experiments of some recent investigators, especially Morris W. Travers,<sup>c</sup> A. Gautier,<sup>d</sup> and K. Hüttner,<sup>e</sup> it is very evident that the gases are often produced by chemical changes resulting from the heat used for their expulsion. Thus, hydrogen may be due to reduction of water by minerals susceptible of oxidation at high temperatures, as ferrous silicates; the carbon monoxide to reduction of carbon dioxide in a like manner or by the hydrogen formed by the first reaction; the methane might arise from interaction between water and traces of metallic carbides, etc.<sup>f</sup>

Free oxygen is not reported as one of the evolved gases, and from the nature of the case it should hardly be expected.

Nitrogen, though found in but small relative amount in the free state, is comparatively abundant and easily detectable as ammonia or ammonium salts in certain classes of rocks and was long since thus recognized. H. Rose<sup>g</sup> says that pitchstone gives off ammoniacal water on heating; A. Delesse<sup>h</sup> found ammonia in rocks, and A. Gautier

<sup>a</sup> Pogg. Ann., vol. 95, 1855, p. 60.

<sup>b</sup> Ann. chim. phys., vols., 15, 17, 18, 21, 1878-1880.

<sup>c</sup> Proc. Roy. Soc., vol. 64, 1899, p. 130.

<sup>d</sup> Comptes rendus, vol. 131, 1900, p. 647; vol. 132, 1901, pp. 58 and 189; vol. 136, 1903, p. 16; Ann. chim. phys., 7th ser., vol. 22, 1901, p. 97; Ann. des Mines, 10th ser., vol. 9, 1906, p. 316.

<sup>e</sup> Zeitschr. anorg. Chemie, vol. 43, 1905, p. 8.

<sup>f</sup> For the methods of collecting and analyzing such gaseous mixtures consult Travers, M. W., The Experimental Study of Gases; the above-cited papers of Gautier; and Moureu, Charles, Comptes rendus, vol. 142, 1906, p. 44.

<sup>g</sup> Quantitative Analyse, Finkener ed., p. 673.

<sup>h</sup> Ann. des mines, vol. 18, 1860, p. 151.



later made quantitative tests; H. Erdmann<sup>a</sup> found that ammonia was obtained by acting on various minerals of ancient igneous rocks with a caustic alkali; C. Luedeking and H. A. Wheeler<sup>b</sup> found ammonium sulphate in a barite from Missouri, the presence of which I was able to confirm.

It has been noted in this laboratory on three separate occasions, when series of ores, roofing slates, and eruptive rocks were analyzed, that ammonia, either in the form of chloride or sulphate, or even as free ammonia, was given off on heating. Its appearance was not limited to one or a few specimens of a series, but seemed to be characteristic of all and to be afforded by the unbroken rock as well as by the powdered sample. The precise conditions under which the specimens were collected not being known, it is impossible to affirm positively that the ammonia may not have been due to recent organic contamination of some sort, especially in the case of the slates, but it is believed that a more critical collection of material will not alter the general result. Its amount was sometimes readily determinable by Nesslerization, being as high as 0.04 per cent in some slates. Carbonaceous organic matter was absent from most of these, but doubtless existed in them in their early history. In their case the ammonia was, in part at least, evolved as such, imparting a strong alkaline reaction to the water in the upper part of the tube. The presence of sulphides, fluorides, or chlorides in the rock might cause the ammonia to appear as a sublimate of sulphate, fluoride, or chloride. It has been suggested that ammonia might result from the action of water on metallic nitrides, O. Silvestri<sup>c</sup> having observed a nitride of iron on a lava from Etna.

## 28. SPECIAL OPERATIONS.

The problem often presents itself of ascertaining the composition of that portion of a rock powder which is soluble in special reagents or in a reagent of a particular concentration. No precise directions can be formulated to meet such cases. The procedure must vary with the character of the constituents of the rock and with the object which it is sought to attain, and only in exceptional cases can a separation of this kind be sharp. Much depends on the degree of fineness of the powder and on the length of time it is exposed to the action of the reagent.

### A. DETECTION OF NEPHELINE IN PRESENCE OF OLIVINE.

For confirmation of the microscopic diagnosis, Prof. L. V. Pirsson<sup>d</sup> has indicated a means of detecting nepheline in presence of olivine, as in nepheline basalts, based on the very ready solubility of nepheline, as

<sup>a</sup> Ber. Deutsch. chem. Gesell., vol. 29, 1896, p. 1710.

<sup>b</sup> Am. Jour. Sci., 3d ser., vol. 42, 1891, p. 495.

<sup>c</sup> Gazz. chim. ital. vol. 5, 1875, p. 301.

<sup>d</sup> Am. Jour. Sci., 4th ser., vol. 2, 1896, p. 142.

compared with olivine, when boiled for but one minute with a sufficiency of very dilute nitric acid (1:40). Gelatinization of the filtrate on evaporation is taken as evidence of the presence of nepheline. If olivine is present in quantity, however, this test must not be accepted at once as final, for some, if not all, olivines are much more soluble in nitric acid of the above strength than Professor Pirsson was led to believe from his original tests. If, therefore, on evaporation of the filtrate, much iron is indicated, the gelatinization may well be due to olivine alone or in part, and then the quantitative relation of silica to iron plus magnesium should be ascertained. It must also be borne in mind that any other very soluble silicates present will be more or less affected, and that apatite is largely or wholly dissolved. It is possible that still more dilute nitric or perhaps some other acid may exert a slighter solvent action on olivine without being appreciably less effective in dissolving nepheline, etc. In combination with a quantitative analysis of the extract the method is perhaps susceptible of a wider application than the particular case for which it was first used. It is well worth further study.

#### B. DETERMINATION OF SOLUBLE SILICA.

Very often in treatment by acids silica is separated in gelatinous or granular form mixed with the unattacked minerals, and it becomes necessary to remove or estimate this silica, or else to discriminate between soluble and insoluble silica already existing together. Usually a boiling solution of sodium carbonate has been employed for this purpose, though the caustic alkalies have found advocates.

G. Lunge and C. Millberg<sup>a</sup> have conclusively shown that quartz is not nearly so insoluble in solutions of the caustic alkalies as has been supposed, but that given a sufficient degree of subdivision it can be brought wholly into solution; that it is impossible to secure correct separation of quartz and opaline silica by the use of either caustic or carbonated alkalies; and that digestion on the water bath for fifteen minutes with 5 per cent solution of sodium carbonate is the only way to secure exact separation of unignited precipitated silica from quartz, and then only when the finest flour has been removed by levigation. The authors say:

If, however, no more of such flour is present than is produced in the ordinary operations of powdering and sifting through cloth of the finest mesh, the error arising from the above-mentioned treatment is so slight that it can generally be neglected; it reaches 0.1 to, at the most, 0.2 per cent of the total silica, by which amount the quartz will appear too low, the amorphous silica too high.

The above authors also show, however, that the solvent action of the caustic alkalies on quartz becomes very apparent only when the material has been reduced to such an utterly impalpable degree of

<sup>a</sup> Zeitschr. angew. Chemie, 1897, pp. 393, 425.

fineness as is practically never reached in the preparation of samples for rock analysis. For this reason I have no hesitation in recommending the employment of a dilute solution of sodium hydroxide when the silica separated by acid from one of several mineral constituents of a rock is to be estimated. Even when dilution is considerable, solution is almost immediate, and as soon as this is accomplished—the point being known by the change in appearance of the residue—the solution should be diluted with cold water and filtered at once. The difficulty met with in filtration may often be overcome by faintly acidifying, which has the added advantage of at once arresting any further action of the alkali. If the dilution is sufficient no separation of silica results from so doing. Very dilute acid should also be used for washing. Lunge, when using sodium carbonate, washes with hot carbonate solution to which alcohol has been added, thus obtaining clear filtrates.

B. Sjollema <sup>a</sup> regards diethylamine as suitable for the separation of amorphous silica from quartz and silicates in soils. He found that a 33 per cent boiling aqueous solution of the reagent dissolved 0.65 gram of amorphous silica practically wholly after eight hours action and a 16 per cent solution within 1 per cent, whereas the solubility of quartz powder was only 1.2 per cent after two days.

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<sup>a</sup> Jour. landw. Chemie, vol. 50, 1902, p. 371; abstracts in Jour. Chem. Soc., vol. 84, 1903, p. 241; Analyst, vol. 28, 1903, p. 123; Chem. Zeit., Rep., 1903, p. 21.

# CARBONATE ROCK ANALYSIS.

## PART I.—INTRODUCTION.

### 29. QUALITATIVE COMPARISON OF CARBONATE AND SILICATE ROCKS.

The analysis of an ideally pure carbonate of any one metal is of great simplicity. Occasionally the carbonates met with in nature approach but probably never fully reach this condition of purity, as when we find them in the form of well-crystallized mineral species, like calcite, magnesite, siderite, cerussite, etc. Even in such cases it is usually true that the crystals either contain included foreign matter to a slight extent or are composed of more than one carbonate in isomorphous mixture, and then the separation of the different metals may demand considerable skill and time. The great carbonate formations of the earth's crust are never of this simple character, however, even the most dazzling white marble contains other ingredients than calcium carbonate, and the degree of admixture may vary from almost nothing up to that indefinite point beyond which the rocks cease to be classified as limestones or dolomites, but rather as calcareous shales, sandstones, etc.

The matters other than carbonates which these rocks may contain are in the main original constituents, having been introduced at the time the carbonates were themselves deposited. They may consist of the detritus of earlier geologic formations in the form of sand or clay that has undergone little or no alteration in the subsequent passage of time, besides carbonaceous matter derived from the marine life existing during their formation. But besides these there may be found secondary minerals that have resulted from chemical transformations in the mass of the accumulated sediments, either with or without the powerful aid of dynamic forces like long-continued movements of the crust or the intrusion of hot igneous rocks. The metamorphism produced by these last agencies is often of a most profound character and may result in the production of a great variety of minerals altogether foreign to the original sediments. The metamorphism may have been accompanied by the introduction and incorporation of new material, but even in the contrary case all the chemical constituents of the original detrital foreign matter have necessarily become a part of the carbonate rocks. These constituents are furthermore necessarily those common to silicate rocks of the earth's crust, hence the carbonate

rocks differ qualitatively in no essential respect from their silicate associates except as to the more common occurrence of carbonaceous matter; and a complete analysis of one of them involves most of the determinations and separations described in the first part of this work. The analytical procedure differs, however, in some respects, owing to the great preponderance in most cases of components easily soluble in the common mineral acids, which either renders it unnecessary to use an alkaline flux or enables the analyst to reduce its amount very materially, thereby simplifying subsequent operations. The analysis is further simplified by the occurrence in reduced amounts, as a rule, of those elements which most complicate the analysis of a silicate rock. Only in the very purest carbonate rocks are some of them nearly or entirely lacking, as silicon, aluminum, and the alkalies, but the minor constituents zirconium, barium, and strontium are often not present in readily determinable amount.

In the following pages the descriptions will apply especially to those large deposits of carbonate materials denominated limestones, magnesian or dolomitic limestones, and dolomites, many of which are of great and growing economic importance. For the purpose of the analyst an exact definition of these terms is of minor significance. Indeed, among geologists there has been no uniform rule for their delimitation, and from the necessity of the case any rule must be purely arbitrary, since there is absolutely no break in the series between limestones and dolomites. The last name strictly applies only to the equiatomic double salt of calcium and magnesium,  $\text{CaMg}(\text{CO}_3)_2$ , and by some it is held that this should be its only application. On the other hand, the opposite extreme has been approached in contending that all limestones with as much as 1 or 2 per cent of magnesium carbonate should be classed as dolomites. According to H. Ries<sup>a</sup> the most prevalent practice among geologists seems to be to call all limestones with from 5 to 18 per cent of magnesium carbonate magnesian limestones and the rest limestones or dolomites, according as their magnesium-carbonate contents lie below or above these limits. Some, however, while subscribing to the above definition of magnesian limestones, would and do call those rocks with from 20 per cent of magnesium carbonate up to the dolomitic ratio dolomitic limestones.

It has seldom been considered necessary to analyze the carbonate rocks with any great attention to detail. The majority of analyses have been made with reference to their technical utilization and with determination of only the more important constituents. In such cases it has been a common practice to omit direct determination of the carbon dioxide and either to calculate its supposed amount on

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<sup>a</sup> Bull. New York State Mus. No. 44, 1901, p. 644.

the basis of the total lime and magnesia found or to regard the loss on ignition as representing the carbon dioxide. The portion insoluble in hydrochloric acid has been frequently stated as silica. Any iron that may have been determined has been reported as  $\text{Fe}_2\text{O}_3$ , water and carbonaceous matter have been entirely overlooked or neglected, also titanium, phosphorus, and the rarer constituents, and sulphur has been almost uniformly reported as  $\text{SO}_3$ . Such practice has repeatedly involved very gross errors of omission as well as of commission, which, though of little moment in many cases from the commercial point of view, detract much from the value of all these analyses in the eyes of the geologist intent on gaining an exact knowledge of an important formation and of the possible sources of metallic ore deposits of economic value, whether these last are residual soils resulting from the bacterial decomposition of limestone formations or whether they are deposits of the ores of lead, zinc, vanadium, etc., that have been formed beneath the surface by the agency of circulating waters.

### 30. MINERAL COMPOSITION OF CARBONATE ROCKS.

The chief mineral compounds entering into the make-up of the carbonate rocks are of course calcium carbonate for the limestones proper, with the double carbonate, the mineral dolomite, in increasing proportions through the magnesian limestones and dolomites; but it is the exception to find them free from other carbonates as essential components, chiefly those of iron and manganese, which are usually in isomorphous combination with the constituents of one or the other of the above minerals. The proportion of these is far less than that of the others, but sometimes aggregates several per cent. When they predominate the rock is no longer a limestone, but becomes an ore of iron or manganese.

Neglect to take account of these minor carbonate constituents is a frequent source of error in the statement of analyses, where it is very common to find reported as carbonates only calcium and magnesium, and perhaps a considerable amount of iron as  $\text{Fe}_2\text{O}_3$  and manganese as one of its higher oxides, although it is practically certain that these must have existed in large part as ferrous and manganous carbonate. A careful determination of the carbon dioxide will often tell whether this is so or not, for it will perhaps be found in excess of that required for lime and magnesia. A negative result, however, is not to be taken as proof of the absence of iron or manganese carbonate or both, for very often a small portion of the magnesia may be in silicate combination, or in some formations there may be a little gypsum to claim a portion of the lime. This last is not likely to be the case in the average hard rock that is used for the manufacture of cement. In such cases sulphur often reported as  $\text{SO}_3$  is likely to come from iron pyrites and perhaps to a limited extent at times from carbonaceous matter.

It is hence easy to see that except in absence of iron and manganese it is not possible to ascertain the exact proportions of calcium and magnesium carbonates, and even then sometimes only after separate analyses of the parts soluble and insoluble in dilute acid have been made.

The commonest sulphide in limestone formations is pyrite, though its presence is often obscured by its extremely fine state of division or by carbonaceous matter. In the metamorphosed limestones, especially those altered by intruded igneous bodies, other sulphides, as sphalerite and galena, are not uncommon, and these and still other sulphides may form important ore bodies at times or again give rise by oxidation to equally or even more valuable ores of a different sort. The strongly metamorphosed siliceous limestones are, however, seldom subjected to chemical analysis, especially for commercial purposes.

The siliceous components may be of the most varied character. Their names alone might fill a page. Suffice it to mention for the metamorphosed rocks garnet, vesuvianite, wollastonite, tremolite, diopside, the scapolites, tourmaline, apatite, and silica in the form of chert. Cherty limestones are extremely common and their silica was doubtless introduced during metamorphism. The chief siliceous components of unmetamorphosed limestone rocks are, as said before, sand and a clay. The latter particularly, since it is a clay, carries water, and this fact is usually overlooked in ordinary analyses. The analytical statements may show no water, though clayey matter is present to a large extent. The sand may be largely or altogether quartz, or it may be composed in part of such other minerals as are commonly found in sands, like mica, garnet, zircon, etc. These latter may be often detected, even in the less siliceous and clayey limestones, by dissolving large amounts in hydrochloric or acetic acid and subjecting the residue to microscopic examination, after burning off obscuring carbonaceous matter, if necessary. The unignited residue will, needless to say, contain the pyrite of the rock and any other sulphides not attacked by dilute acid. In the case of some nonsiliceous rocks it may even consist to a considerable extent of iron oxide, probably in most cases one of the hydrated oxides, but perhaps often hematite. The presence of such a constituent in a limestone is occasionally indicated by a reddish cast of color in the fresh rock, provided there is not enough carbonaceous matter to obscure it. The existence of a ferric oxide is not always incompatible with the simultaneous presence of this carbonaceous matter.

A purple color may be due, as in some slates, to admixture of a ferric oxide or hydroxide with carbonaceous matter, while to the latter component are due the gray and black colors often encountered. The color of greenish sedimentary limestones may be caused by certain minerals of the mica family, as the chlorites.

The condition of phosphorus when it occurs in only minute amounts is not determinable with certainty. It may sometimes doubtless be found in traces in the form of heavy phosphates like monazite and xenotime in the insoluble residues, and at others it is held by iron and aluminum, but in the highly phosphatic rocks its usual form is overwhelmingly that of phosphorite or apatite, which may, in fact, become the predominating constituent.

It is known from my analyses that vanadium is a regular though very minor constituent of limestones as well as of sandstones and igneous rocks generally. Recent tests on 100-gram lots of a great limestone formation in northwestern Mexico show that it at least carries molybdenum as well as vanadium, and also nickel. Nothing is known with certainty regarding the mode of combination of these elements in limestones, though it is quite possible, as to the vanadium at least, that it was an original ingredient of the aluminous silicates of the clayey matter, and that nickel, zinc, and lead when present are mainly in the form of carbonate or sulphide.

Carbonaceous matter has been frequently alluded to as a common constituent of secondary importance. It is absent from the white marbles and some colored decorative stones, but forms an appreciable percentage of other limestones in extreme cases.

Some kinds of bituminous limestone are characterized by a fetid odor when struck or abraded. This is perhaps due in most cases to hydrogen sulphide, either original or resulting from decomposition of organic matter. The presence of hydrogen sulphide is sometimes unmistakable, and Prof. B. J. Harrington has determined its amount at about 0.02 per cent in the form of liquid inclusions in a fetid calcite from Canada.

The above remarks do not exhaust the subject of the mineral composition of limestone rocks, this term being used here and elsewhere in its generic sense to include the magnesian varieties. They may serve as a sufficient guide, however, to those undertaking their analysis. Ignorance of the mineral composition in respect to the minor components has been a prolific source of error on the part of those analyzing these rocks.

### 31. BEHAVIOR TOWARD DIFFERENT REAGENTS AS A MEANS OF DISTINGUISHING DIFFERENT CARBONATES.

Although for the analyst provided with laboratory facilities it is usually of little importance to possess other means for identifying qualitatively the different varieties of carbonate rocks than the conventional methods of qualitative analysis, some of the rapid methods for so doing may serve a most useful purpose at times, especially in the field, where transport of bulky apparatus and quantities of liquid reagents is difficult or impossible.



## a. DIFFERING SOLUBILITY IN TARTARIC AND CITRIC ACIDS AND IN ACID POTASSIUM SULPHATE.

This subject, touched on now and then by earlier writers, was first studied by H. C. Bolton,<sup>a</sup> and many of his results were long afterwards confirmed by J. W. Richards and N. S. Powell.<sup>b</sup> Solid organic acids like citric and tartaric were found to be well adapted for distinguishing between many carbonate minerals by the difference in the degree of action on them. Potassium acid sulphate had earlier been used to some extent by E. Jannettaz,<sup>c</sup> and Richards and Powell give data as to this reagent for carbonates. It will suffice here to refer to the originals, especially Bolton's papers, where will be found data as to the solubility of a great variety of minerals in the above-named solvents as well as in others of inorganic nature.

For the present purpose it is more important to present in brief the methods proposed for distinguishing between calcite and aragonite and between relatively pure limestones and those high in magnesium.

## b. THE MEIGEN REACTION FOR DISTINGUISHING CALCITE FROM ARAGONITE.

According to W. Meigen,<sup>d</sup> calcite powder when boiled with a dilute solution of cobalt nitrate remains white or, in presence of organic matter, becomes yellowish, while aragonite turns lilac red from formation of a basic cobalt carbonate. G. Panebianco<sup>e</sup> obtained different results. Calcite after a minute's boiling with the cobalt nitrate reagent became sky blue and after four minutes lavender blue. Aragonite became at once lilac and later violet, the difference in the colors being supposedly due to different basic carbonates. Panebianco also observed that the reaction was useless for proving absence of calcite when mixed with aragonite, for the color characteristic of the latter still appeared when 19 parts of calcite were mixed with but 1 of aragonite. Freshly precipitated gelatinous calcium carbonate, as well as that from warm solutions, gives the aragonite reaction, but that formed at 0° affords the calcite reaction.

A. Hutchinson<sup>f</sup> seems to have shown that the disagreement between the results of these investigators is to be attributed to the presence of iron in the cobalt nitrate used by Meigen.

According to Meigen barium and strontium carbonates, but not that of magnesium, behave like aragonite, and calcium phosphate produces a blue powder.

<sup>a</sup> Ann. New York Acad. Sci., vol. 1, 1877-1880, pp. 1, 153; vol. 2, p. 1; Proc. Am. Assoc. Adv. Sci., vol. 31, 1883, p. 271; Chem. News, vols. 36, 37, 38, 43, 47, 1877-1883; Rept. Brit. Assoc. Adv. Sci., 1880, p. 506; Min. Mag., vol. 1, 1880-81, p. 181; Ber. Deutsch. chem. Gesell., vol. 13, 1880, p. 726.

<sup>b</sup> Jour. Am. Chem. Soc., vol. 22, 1900, p. 117.

<sup>c</sup> Comptes rendus, vol. 77, 1873, p. 838; vol. 78, 1874, p. 852.

<sup>d</sup> Centralbl. Min., 1901, p. 577.

<sup>e</sup> Rivista min. crist. ital., vol. 28, 1902, p. 5.

<sup>f</sup> Min. Mag., vol. 13, 1903, p. xxviii.

## C. DISTINGUISHING BETWEEN CALCITE AND DOLOMITE.

It must be understood that magnesian and dolomitic limestones represent, as a rule, mixtures of the double carbonate dolomite with calcite, and not of calcite with magnesite. Calcite is easily soluble in dilute hydrochloric and acetic acids; dolomite and magnesite are nearly insoluble. Hence calcite effervesces strongly with these acids, even in lump form, while dolomite shows little or no effervescence. An easy means is thus afforded for distinguishing the two. Dolomitic limestones are also but feebly attacked, according to their content in calcite, so that in powder form it is not difficult to make a fair separation of calcite from dolomite and magnesite, but not from magnesian hydrocarbonates, which last, however, are never essential components of the great carbonate formations.

J. Lemberg<sup>a</sup> found that calcite responds by the formation of a violet color in its powder when treated with a solution of aluminum chloride and hematoxylin (extract of logwood), but that dolomite remains unaffected. The solution is prepared by dissolving 4 parts of dry aluminum chloride in 60 parts of water, adding 6 parts of logwood, and boiling for twenty-five minutes with replacement of the escaping water. The deep-violet solution is then filtered.

Coarsely powdered Iceland spar and Carrara marble became violet after five to ten minutes immersion and subsequent removal of the adhering solution with water. In the powder of a dolomitic limestone the calcite grains were easily distinguishable from those of dolomite by this means. It is not advisable to prolong the treatment unnecessarily, since after twenty minutes the dolomite surfaces may show occasional pale-blue spots. The reaction is caused by precipitation upon the calcite of aluminum hydroxide, which forms, with the coloring matter, a lake.

F. Hinden<sup>b</sup> finds that limestone (calcite) at room temperature gives carbon dioxide with 10 per cent solution of ferric chloride, but dolomite does so only on warming. A second reagent mentioned by him is copper-sulphate solution, from which basic copper carbonate is separated by calcite, but is not by dolomite.

The behavior toward heat alone is sometimes useful as a distinguishing test, for dolomite loses its carbon dioxide so easily that the powder is violently projected from the crucible if the heat is not very cautiously applied, whereas calcite can be strongly heated at the start without danger of loss of solid matter.

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<sup>a</sup> *Zeitschr. Deutsch. geol. Gesell.*, vol. 40, 1888, p. 357.

<sup>b</sup> *Verh. Naturforsch. Gesell. Basel*, vol. 15, 1905, p. 1.

## PART II.—REFINED METHODS OF ANALYSIS.

## 32. SILICA, ITS SEPARATION FROM ALUMINA, ETC.

## A. METHODS OF DECOMPOSING THE ROCK.

Few limestones are so pure that they leave no residue on treatment with hydrochloric acid. The residue may consist of quartz, clay, or other silicates, carbonaceous matter, pyrite, etc., and the mode of attack employed will depend on the amount of insoluble matter, and whether or not it is desired to know its composition separately from that of the soluble portion.

## a. WHEN THE INORGANIC RESIDUE IS TO BE SEPARATELY ANALYZED.

A gram of the rock powder is moistened with water and dissolved in dilute hydrochloric acid (or acetic, if the former acid is likely to act appreciably on the silicates) in a covered beaker till all effervescence ceases. Moderate heat is needed if the effervescence is so weak as to indicate a rock of dolomitic character. The solution is filtered through a 7 cm. filter and the residue washed with water, or hot dilute hydrochloric acid should gypsum be present and the silicates resistant. The paper, with its contents, is ignited moist in platinum (blast needed only if the amount is considerable), and after weighing it is fused with sodium carbonate and analyzed like a silicate rock (d. p. 71 et seq.). If it contains hydrous minerals the original water content of the residue is best determined by a separate test on a fresh portion of the sample, and not by drying and weighing on a counterpoised filter and then igniting. The weight of water found should be added to that of the ignited residue in order to get the true weight of the insoluble matter.

If the ignited residue is wholly quartz, or is very insignificant in amount, it may be treated at once with a drop of sulphuric acid and a few drops of hydrofluoric acid, and the acids removed in the radiator shown in fig. 2 (p. 29). If then a slight residue still is visible, it is well to repeat the treatment with acids and evaporation, for it can not be too insistently pointed out that quartz resists the action of hydrofluoric acid far more than many silicates, and several evaporations may be needed to volatilize the crystallized mineral unless it has first been reduced to a most impalpable state of division. When the weight after exposure for a few minutes to the full burner heat no longer changes, the loss represents silica. If a slight residue still shows (it is most likely to be mainly alumina), the subsequently obtained precipitate of alumina, etc., is added to it and ignited in the same crucible. The filtrate is best treated as in 33. A. a, page 178.

If the original residue is small, and accurate knowledge of its composition is desired, several grams of the rock may be dissolved and the

whole filtrate best treated for dissolved iron, aluminum, and manganese, as in 33. A. a, page 178, but only an aliquot part of the subsequent filtrate should then be used for the determination of calcium and magnesium.

In case the filtrate from the residue is perceptibly colored by dissolved organic matter, a condition that may occasionally arise, this organic matter must be effectually destroyed before proceeding to the precipitation of iron and aluminum, for otherwise incomplete precipitation of one or both will be the result. The complete removal of this disturbing material can not always be effected by ordinary oxidizing agents, but only by evaporation and heating to its carbonization point. In doing this it is necessary, in order to avoid possible loss of iron as chloride, to evaporate with nitric acid to dryness a couple of times and then to heat over a free flame gently till the desired result is achieved. If this seems undesirable for any reason, it may be better to start with a fresh portion of the limestone and to treat it by one of the methods under b, below, for the bulk analysis.

**b. WHEN THE INORGANIC RESIDUE IS NOT TO BE SEPARATELY ANALYZED.**

This will be the usual case. As said above, the residue may consist of clay or other silicates, carbonaceous matter, pyrite, etc., with or without quartz. There are two ways open to render the non-combustible part of this soluble.

*α. By solution in acid after strong ignition.*—This is the best method to employ in all cases where the ratio of insoluble to soluble compounds is not less than is found in an argillaceous limestone that is directly suited for burning to Portland cement—that is to say, when the silica does not much exceed 15 per cent and the oxides of iron, aluminum, and titanium together are not in excess of 6 per cent. The exact allowable limits have not yet been determined, nor is it known what the proportions may be in dolomites and dolomitic limestones. This is a subject for further investigation.

Limestones, however, in which the above percentages of silica, alumina, etc., are not exceeded may be converted in ten to fifteen minutes by a good blast capable of giving a temperature of 1,100° to 1,200° to a product that is wholly soluble in hydrochloric acid, provided the rock was first reduced to a very fine powder.

A gram of the powder is heated in a covered platinum crucible by an inclined blast. If a limestone, the flame may be applied at once, as a rule, without fear of loss, or after short exposure to a full Bunsen flame. Highly magnesian limestones, if this method is applicable to them, must, however, be heated with the greatest caution, for their temperature of decomposition is far below that of limestones, and violent projection of material often begins far short of visible redness.

The strong heating is usually stopped after ten or fifteen minutes, when the shrunken product may have the appearance of a sintered or even clinkered mass that detaches for the most part readily from the crucible.<sup>a</sup> It is transferred to a beaker or evaporating dish and moistened with water. The crucible is then cleaned with hydrochloric acid (1:1) and the contents are poured into the beaker or dish. By gentle heat and cautious pressure with the flattened tip of a rod the lump or lumps are caused to disintegrate and pass largely into solution in a few minutes. A certain amount of silica may remain undissolved in a flocculent state, but this is of no moment. When all grit has disappeared, the liquid, if in a beaker, is washed into a dish and evaporated to dryness. If solution was made directly in the dish the volume of liquid need not exceed a few cubic centimeters and the evaporation takes but a short time. A much smaller dish is allowable than in the case of wholly siliceous minerals, because of the small bulk of solution and the entire absence of added fixed salts.

*β. By solution in acid after heating with sodium carbonate.*—When the siliceous components are in such amount as not to permit the formation of a wholly soluble product by strong ignition, it is necessary to mix sodium carbonate with the powder and decompose the silicates by its aid over the blast lamp. This procedure may be adopted with all siliceous limestones if desired. The amount of flux to be used is but a fraction of that needed for a silicate analysis. One-fourth to one-half gram for 1 gram of limestone suffices,<sup>b</sup> for the lime formed by ignition is itself a powerful flux and it is quite unnecessary to have here a liquid fusion. A sintering suffices. This proportion holds also for highly magnesian limestones and for cement rocks containing as little as 50 per cent of lime.<sup>c</sup>

#### B. SEPARATION OF SILICA.

In this connection the remarks under *β*, page 76, should be carefully read.

Because of the absence of large amounts of alkali salt and of much silica the evaporation of the hydrochloric-acid solution obtained in A. a. and b. *α*, and *β*, takes little time as compared with a silicate analysis. When dry, or nearly so, on the steam bath, the dish may be placed in an air bath or (covered) on a platinum triangle resting on a hot plate, and heated to 200° for an hour if a limestone is under treatment, but for a highly magnesian limestone

<sup>a</sup> The changes that take place during the ignition comprise loss of all carbon dioxide, water, and carbonaceous matter; oxidation of all pyrite, with retention of the whole of the sulphur as calcium sulphate. Prolonged heating will gradually expel all the sulphur trioxide from the calcium sulphate and later the alkalis, which can be wholly volatilized in an hour or less by a powerful blast. With an inclined blast the alkalis condense in part on the under side of the lid as a soluble and powerfully alkaline deposit, sometimes weighing several milligrams, but in the time above set no loss of alkali appears to occur.

<sup>b</sup> Shimer, P. W., in Meade, R. K., *The Chemist's Pocket Manual*, p. 162.

<sup>c</sup> Personal communication from R. K. Meade.

the temperature should not exceed  $120^{\circ}$ , according to J. P. Gilbert,<sup>a</sup> because of recombination of silica and magnesia, with subsequent re-solution of the silica when acid is added.

Bertram Blount claims<sup>b</sup> that by heating to  $200^{\circ}$  in this manner the silica is completely separated by a single treatment, but I have not found it so, though the amounts going into solution on subsequent addition of acid are small, seldom over 2 or 3 milligrams. Therefore, for exact work with silica present to the amount of 2 to 4 per cent and over, that already rendered insoluble should be filtered off and the solution again evaporated. With this end in view the dry mass is drenched with strong hydrochloric acid and allowed to stand for a few minutes, then as much water is added and the dish covered and placed on the bath for ten minutes. Instead of strong acid that of half strength may be used and the heating begun at once. The silica is then separated by filtration on a filter of suitable size, washed thoroughly with dilute acid and then twice with cold water. The filtrate is evaporated again to dryness, the residue extracted with hydrochloric acid as before, but with allowance of only a few minutes' time, and the solution filtered once more through a second and smaller paper. The two papers with their contents are slowly dried, charred, and ignited in platinum, finally over the blast for ten minutes. The weighed silica is to be corrected for foreign matter by evaporating with 5 cm.<sup>3</sup> of hydrofluoric acid and one or two drops of sulphuric acid, as directed in c (p. 79). The residue obtained after ignition is almost always considerably less than that found in analyzing a silicate rock and an ignition of a minute or two over a full burner, after expulsion of the sulphuric acid, is in almost all cases sufficient. The composition of this residue is similar qualitatively to that derived from silicate rocks. It should never contain calcium or magnesium, but consists mainly of alumina, with a little ferric, titanic, and phosphoric oxides. It is preserved and the subsequently obtained precipitate of these oxides is added to it and ignited in the same crucible.

33. ALUMINUM, TOTAL IRON, TITANIUM (SILICA, MANGANESE); PRECIPITATION IN COMPANY WITH PHOSPHORUS, ETC.<sup>c</sup>

The next steps differ widely according to the relative amounts of iron, aluminum, etc., taken collectively, and according as it is the intention to precipitate manganese with these or after separation from them. The individual preferences of experienced analysts will often differ as to the selection of the particular method, and it is not intended here to prescribe definitely the course that anyone should

<sup>a</sup> Technology Quart., vol. 3, 1890, p. 61; abstract in Zeitschr. anal. Chemie, vol. 29, 1890, p. 688.

<sup>b</sup> Jour. Soc. Chem. Ind., vol. 21, 1902, p. 1217.

<sup>c</sup> See also 8, p. 81.

follow. Nor in the following are all methods of separation that may be capable of affording good results noticed. There are some that seem promising but that have not yet been studied sufficiently in this laboratory, nor in fact elsewhere, to justify description. A method that may be well adapted to a certain combination of elements may be valueless if to that combination a single other element is added; or it may be that the possible disturbing influence of that element has not been as yet ascertained.

#### A. PRECIPITATION OF ALUMINUM, IRON, ETC.

##### a. COLLECTIVE PRECIPITATION OF ALUMINUM, IRON, MANGANESE, TITANIUM, AND PHOSPHORUS BY AMMONIUM SULPHIDE.

If these elements collectively represent 2 or 3 per cent or less of the rock, the filtrate from the silica (32. B, p. 176), reduced in bulk if need be, is put into a flask of 150 cm.<sup>3</sup> capacity with enough hydrochloric acid to prevent precipitation of magnesium when made ammoniacal. This last is a most important point. Ammonia free from carbonate is then added to strong alkalinity and hydrogen sulphide is introduced to saturation, followed by a fresh addition of ammonia equal to the excess first used. Boiled water is then poured in till well up in the neck, the cork is inserted, and the flask is set aside for twelve to twenty-four hours. Its contents are then collected on a filter of small size (7 cm.) and washed with water containing a little ammonium sulphide and nitrate. Precipitation is not absolute as to one or more of the constituents of the separated mixture, but the amounts recoverable from the filtrate are usually very minute. (For treatment of the filtrate see 36, p. 183.)

The paper and its contents, having been washed with ammonium nitrate, may be safely charred and ignited as they are in the crucible containing the residue left after volatilizing the silica. The final weight after ignition in full contact with air, followed by application of the inclined blast for not over five minutes, represents  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{Mn}_2\text{O}_3$ ,  $\text{TiO}_2$ , and  $\text{P}_2\text{O}_5$ . (For the separation of these, see C, p. 180.)

This method is inapplicable for phosphatic limestones containing insufficient aluminum to more than combine with the phosphorus, for then calcium phosphate would be precipitated also. In such case method b or c would apply if the iron and aluminum together more than sufficed to carry down the phosphorus, but in general it would be necessary to add a known amount of pure iron oxidized to the ferric state, then to precipitate by ammonia or the basic-acetate process, and to deduct the added iron oxide from the weight obtained.

**b. PRECIPITATION OF ALUMINUM, IRON, TITANIUM, AND PHOSPHORUS BY AMMONIA.**

The filtrate from silica is boiled in a beaker of good glass, with addition of a few drops of bromine water or two or three of strong nitric acid, till all trace of bromine or chlorine is gone. Then hydrochloric acid is added, if not already present, in amount sufficient to prevent precipitation of magnesium when rendered ammoniacal. Ammonia is added to the boiling solution (in platinum if possible), 100 to 200 cm.<sup>3</sup> in volume, in slight excess, and the boiling is continued for two or three minutes. The precipitate is allowed to settle in the covered vessel, collected quickly on a filter, washed with hot water two or three times, and sucked dry. It is then redissolved in hot and moderately strong hydrochloric acid, the solution is boiled to drive out the trace of chlorine derived from solution of any precipitated manganese dioxide, and the precipitation is repeated just as described once, twice, or even three times, according to the amount of manganese in the rock. Macerated filter paper is added before the last precipitation if the oxides are in large amount. The final precipitate is washed with water containing a little ammonium nitrate. (For treatment of the filtrate see B, below.)

The precipitate is ignited moist in the crucible containing the residue from volatilization of the silica by hydrofluoric acid, and the combined weights increased by traces subsequently recovered (B, below) represent all iron as  $\text{Fe}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{TiO}_2$ , and  $\text{P}_2\text{O}_5$ . (For separation of the ignited oxides see C, p. 180.)

**c. PRECIPITATION OF ALUMINUM, IRON, TITANIUM, AND PHOSPHORUS BY THE BASIC-ACETATE METHOD.**

This method of separation is less employed in limestone analysis than in that of silicate rocks, by reason of the relatively smaller amounts of those elements that precipitate in the basic-acetate process, and hence of their more ready separation by ammonia from appreciable quantities of manganese. (For its application, see A. b.  $\beta$ , p. 83, giving due regard to the smaller amounts of iron, etc., involved in the case of limestones.)

**B. TREATMENT OF THE FILTRATES FROM ALUMINA, ETC.**

The combined filtrates obtained in A. b (or c), above, which, besides all the calcium and magnesium, will contain the manganese and usually traces of aluminum and even iron, are evaporated to a small bulk in platinum with the addition of a few drops of ammonia from time to time, and any precipitate that separates is collected. This is washed a little, redissolved in hydrochloric acid (with addition



of a drop or two of sulphurous acid if its color indicates precipitation of some manganese), and reprecipitated by ammonia at boiling heat in a very small beaker. If need be the precipitation is to be again repeated, but this will seldom be the case. A first precipitation is, however, called for in order to separate a little calcium carbonate that may have precipitated during the evaporation of the original ammoniacal filtrate. The alumina and perhaps trace of iron oxide thus obtained are added to that already found (A. b, c, p. 179). (For treatment of the filtrate, see 34, p. 181.)

#### C. SOLUTION AND SEPARATION OF THE OXIDES OBTAINED IN A AND B.

The same course is followed, whether method a, b, or c under A, pages 178–179, has been employed for precipitating aluminum, iron, etc. According to the weight of the ignited oxides, these are fused with from one to several grams of potassium or preferably sodium pyrosulphate<sup>a</sup> in the covered crucible over a low flame. By using pyrosulphate instead of acid sulphate the time of fusion is greatly shortened, especially when the oxides are in a finely divided state, as after precipitation in presence of macerated filter paper.

When decomposition is complete the cooled melt is dissolved in hot dilute sulphuric acid, the solution is transferred to a capacious platinum dish and evaporated as far as practicable on the bath. Strong sulphuric acid is cautiously added, and evaporation is continued over a radiator, or on a sand bath, or by aid of a free flame applied with care to prevent spattering, till fumes of acid appear freely. When cool the melt should be rather pasty and not hard and dry. It is dissolved by the aid of heat in a moderate amount of water and the solution is allowed to digest for ten or fifteen minutes on the bath. A few flocks of silica will then generally be apparent at the bottom of the dish. Whether visible or not, the solution is passed through a 7-cm. filter, which is then washed with hot water till free from salts.

##### a. SILICA (BARIUM).

The paper with the silica is ignited, weighed, and then treated with a drop of sulphuric acid and a few drops of hydrofluoric acid and the crucible is again weighed. The loss represents silica that escaped earlier separation, and its amount is to be added to that already found.

If a final residue is reddish, it is to be brought into solution by a little pyrosulphate and its solution, if clear, added to the main filtrate containing the mass of the iron. If not clear, the turbidity may be due to a trace of barium sulphate, which should of course be separated and deducted from the weight of the alumina plus other oxides.

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<sup>a</sup> For its preparation see p. 35.

## b. IRON.

In this solution the iron is to be determined by potassium permanganate after reduction by hydrogen sulphide, exactly as detailed under 8. B. a, page 90. The amount of vanadium present is, so far as known, always so small as to make it unnecessary to follow the procedure under 8. B. b, page 92.

## c. TITANIUM.

After titration of the iron the solution is treated exactly as prescribed under 8. B. a.  $\gamma$ , page 92, and the titanium is determined colorimetrically (p. 110).

## d. ALUMINUM.

The aluminum is determined by difference after subtracting the weights of iron and titanium oxides found as above, and also the phosphoric oxide found in a separate portion of the sample (p. 184), and further the manganoso-manganic oxide, if this had intentionally been precipitated along with the iron and aluminum as in A. a, page 178. (For the discussion of the relative merits of this indirect and the direct methods for arriving at the aluminum, see 8. A. a, p. 81, and C, p. 93.)

## 34. MANGANESE.

## A. IN THE FILTRATES FROM 33. B (p. 179).

The filtrates containing the calcium, magnesium, and manganese are caught in a flask of about 150 cm.<sup>3</sup> capacity, 2 cm.<sup>3</sup> of ammonia are added, and hydrogen-sulphide gas is introduced to saturation, then additional ammonia is added to equal that just prescribed. The flask is set aside, stoppered for twelve to twenty-four hours, the precipitate (perhaps colored dark by a little platinum, iron, or other heavy-metal sulphide<sup>a</sup>) is collected on a 7-cm. filter and washed with a dilute solution of ammonium chloride carrying a little ammonium sulphide.<sup>b</sup> (For treatment of the filtrate, see 35, p. 182.)

If the manganese is not to be determined in a separate portion of the rock, the flask is then cleaned from possibly adhering sulphide with a solution of half-strength hydrochloric acid diluted with five times its volume of hydrogen-sulphide water, which is then passed through the filter in order to dissolve the manganese sulphide. The filtrate is treated exactly as under 9. B. b, page 98, if the gravimetric method of determination is chosen. In view of the relatively enor-

<sup>a</sup> More than a negligible trace of iron should not appear at this point. If, however, the organic matter of the limestone is at all soluble in acids and the method of direct solution has been employed, it may very well happen that some iron (aluminum likewise) will fail of precipitation by ammonia. The former will then appear at this stage. Hence, prior ignition of the limestone is imperative in such cases.

<sup>b</sup> This is best prepared by considerably overneutralizing a little hydrochloric acid in a small beaker, and then adding hydrogen-sulphide water or introducing hydrogen-sulphide gas for a few moments, and further diluting if need be.

mous errors affecting this method when small amounts are in question (see 9. A, p. 96), it is far safer to evaporate the hydrochloric-acid solution with nitric or sulphuric acid, till no trace of chlorine is left, and to apply the colorimetric method (9. C. b, p. 99).

If the manganese is to be determined in a separate portion, the sulphide precipitate is discarded; indeed, if it is known that but a few hundredths of a per cent are present, its separation at this stage may be altogether omitted.

#### B. SEPARATE DETERMINATION OF MANGANESE.

Small amounts are always best determined by colorimetry, and then with greatest accuracy in a separate portion of the sample. This, if wholly soluble in acid, is to be dissolved by dilute nitric acid free from hydrochloric acid, the solution filtered if necessary, and further treated, in whole or in an aliquot part, as in 9. C. b, page 99. If not altogether soluble in acid or if the solution is colored by organic matter, it is best to heat the powder with half its weight of sodium carbonate (itself free from manganese) over a strong blast, to digest the cooled mass with nitric acid till any residue that may remain is entirely colorless, to filter into a suitable-sized flask, and to determine as in 9. C. b, page 99.

#### 35. COPPER, NICKEL, COBALT, ZINC, RARE EARTHS, CHROMIUM, VANADIUM, MOLYBDENUM.

It is useless, as a rule, to look for any one of these constituents in the amount of sample taken for the general analysis. From 50 to 500 grams of material will be required. This is dissolved in hydrochloric acid and the residue separated by filtration. If it is desired to examine this residue it is best done by analyzing it separately, after fusion with sodium carbonate and leaching with water. Vanadium and molybdenum will then be found in the filtrate, the other metals in the residue. The former can be determined according to 18, page 126; the latter by the usual methods.

For the hydrochloric-acid solution, which will contain the above elements mainly or wholly, the treatment given under 33. A. a, page 178, is probably best adapted and the following outline may serve as a guide.

The precipitate produced by ammonium sulphide is to be dissolved, the copper and perhaps some molybdenum precipitated by hydrogen sulphide, the filtrate oxidized and evaporated to dryness and the residue boiled with a solution of oxalic acid. The rare earths, perhaps contaminated by silica, will be thrown out and are to be washed with dilute oxalic-acid solution, ignited, treated with sulphuric and hydrofluoric acids, redissolved and reprecipitated in neutral or nearly

neutral solution by oxalic acid, ignited, and weighed. They can then be tested as to their character by methods to be found in special treatises on the rare earths. The filtrates are to be freed from oxalic acid by evaporation and gentle ignition and the residue redissolved and tested for nickel, cobalt, zinc, chromium, vanadium, and molybdenum by approved methods. It must not be forgotten that the filtrate from the original precipitation by ammonium sulphide may hold vanadium, molybdenum, and perhaps nickel, which are to be recovered by acidifying with dilute acetic acid.

Vanadium may usually be found and determined with sufficient accuracy in 5 grams of the limestone by fusing with sodium carbonate and further treating as in 18. C, page 127.

### 36. CALCIUM, STRONTIUM, BARIUM, MAGNESIUM (MANGANESE).

The filtrate obtained in 33. A. a, page 178, or 34. A, page 181, is treated for the most part as in silicate analysis (pp. 101-110).

With dolomitic limestones and dolomites it will be usually impossible to obtain a correction by the method given in C. b, page 109, for the small amount of calcium with the magnesium pyrophosphate. Possibly a better result might be had by adding to the nearly neutral solution of the pyrophosphate a few drops of sulphuric acid and a volume of alcohol equal to that of the solution, in order to precipitate calcium sulphate, but this has not been put to the test in presence of phosphoric acid, though it works well in its absence.

The accurate determination of barium in a limestone is more difficult than in a silicate rock. It is perhaps best to recover it, if present, from the filtrates from the calcium oxalate, after evaporating them and expelling ammonium salts, by taking up the residue in as little hydrochloric acid as possible and, without filtering, adding a few drops of sulphuric acid and allowing to stand for several hours. The washed and ignited precipitate is evaporated with a drop of sulphuric acid and a few drops of hydrofluoric acid to remove silica, the final residue is dissolved by a little hot strong sulphuric acid, and the resulting solution poured into a few cubic centimeters of cold water, the crucible being well rinsed with cold water by the aid of a feather or "policeman." If a precipitate now appears it is barium sulphate, which is collected and weighed in the customary manner.

For the utmost refinement in the determination of the magnesium it is probably best to evaporate the filtrates from the calcium oxalate to dryness, expel the ammonium salts by ignition, add hydrochloric acid and reevaporate, take up again with as little acid as possible, and filter through the smallest filter. The filter is preserved for later use. To the filtrate, which should be very small in volume, a few drops of sulphuric acid are added, and if after some hours a precipitate of barium

sulphate has appeared it is collected and burned in platinum with the previous filter and its contents. The ignited matter is treated as in the preceding paragraph.

To the filtrate is added enough hydrochloric acid to prevent precipitation of magnesium when rendered ammoniacal. Ammonia in slight excess is added and the solution is boiled for some minutes in a platinum dish. There will probably appear a very slight precipitate containing aluminum hydroxide, which is to be redissolved and reprecipitated. In the filtrate the magnesium is precipitated in the manner elsewhere detailed (p. 107).

### 37. PHOSPHORUS.

The rock is dissolved in dilute nitric acid, the solution filtered, the residue fused with a little sodium carbonate, and its nitric-acid solution added to the other.

Or, if the rock is rather argillaceous, the powder may be mixed with half its weight of sodium carbonate and strongly blasted. The nitric-acid solution of the mass is then evaporated to dryness to remove most of the silica, which is collected on a filter, washed, ignited, and evaporated with hydrofluoric and nitric acids. The evaporation with nitric acid alone is repeated once or twice to expel the fluorine, and the residue is dissolved finally in nitric acid and its solution added to the former.

To the solution obtained in either of the above ways ammonium-molybdate solution is added and the precipitate treated by one of the approved methods.

### 38. FERROUS IRON.

The presence of carbonaceous matter in limestones renders the exact or even approximate determination of ferrous iron often impossible. Nevertheless, even in its presence acceptable results are sometimes obtainable if there is not much of such matter and if it does not give with acid a colored solution. But even then it is only the soluble iron, existing chiefly if not wholly as carbonate, that is determinable. Occasionally limestones show films of manganese peroxide, which likewise interferes with the determination.

#### A. IN ABSENCE OF CARBONACEOUS MATTER.

##### a. FERROUS IRON SOLUBLE IN SULPHURIC ACID.

The powder, one to several grams, is introduced into a stout flask of about 200 to 250 cm.<sup>3</sup> capacity and boiled with a little water till all air is expelled. While still boiling dilute sulphuric acid is added a

little at a time, till effervescence ceases, and then a further amount. Calcium sulphate precipitates, but the iron will remain in solution. The flame is then removed and a stopper tightly inserted through which passes a small stopcock funnel. When cool or nearly so, cold water is poured into the funnel, the cock cautiously opened, and the water drawn into the flask, more water being poured into the funnel as fast as it empties, till the solution amounts to 100 or 150 cm.<sup>3</sup>. Such precaution to exclude air is hardly necessary in most cases, however, for in presence of sulphuric acid the oxidation of ferrous iron is exceedingly slow. It is ordinarily quite sufficient to equalize the internal and external pressures by opening the cock, removing the stopper, and pouring in cold water. The flask is then brought under a burette containing dilute permanganate solution and the iron titrated without delay. With a carbonate which is wholly decomposable without the aid of heat, this may be accomplished in a flask filled with carbon dioxide, whereby the danger of attack of silicates is lessened. If the preference is for the potassium-bichromate method of titration, hydrochloric acid may be used instead of sulphuric, in absence of manganese peroxide. In this case there is of course no separation of an insoluble calcium salt, a fact which renders easier the subsequent determination of the iron in any insoluble residue the rock may yield. The ferrous iron thus found is mostly, if not altogether, that existing as carbonate.

#### b. FERROUS IRON IN THE INSOLUBLE RESIDUE.

The titrated solution is filtered through a medium-sized hardened filter, the insoluble matter collected thereon and washed with water. The contents of the paper are rinsed into a capacious platinum crucible, the water mostly evaporated, and in the residue the ferrous iron is determined as in 19. C. d, page 140.

The total ferrous oxide found in a and b, when calculated to ferric oxide and subtracted from the total iron in the same state, gives the ferric oxide in the rock. If separate analyses have been made of the parts soluble and insoluble in dilute acid, similar calculations applied to the iron derived from the two portions will give the ferric oxide in each.

#### c. TOTAL FERROUS IRON.

If separate determinations are not desired, water is poured upon the powder in a capacious platinum crucible, dilute sulphuric acid is slowly added till effervescence ceases, then the crucible is placed in one of the forms of apparatus described in 19. C. d, page 140, together with 5 cm.<sup>3</sup> of hydrofluoric acid, the decomposition of the silicates is effected in an atmosphere of carbon dioxide, and the titration is made as there directed.

**B. IN PRESENCE OF CARBONACEOUS MATTER.**

Decomposition is effected in a flask by dilute sulphuric acid in an atmosphere of carbon dioxide. With limestones and active agitation no heat need be used, but with dolomites it will be necessary. The solution is then quickly filtered through asbestos (in an atmosphere of carbon dioxide if much iron is present), the residue and filter washed a few times, and the filtrate titrated at once with permanganate. If it is colored by organic matter, the result may be in error.

As in A. a, page 184, hydrochloric acid and titration by dichromate may be used in absence of manganese peroxide. In either case it is important to allow the acid to act no longer than is necessary and to filter quickly. A determination of the ferrous iron in the insoluble matter is usually not worth attempting, because of the admixed organic matter.

**39. ALKALIES.**

The alkalies in carbonate rocks are of course constituents of the siliceous components. Since these are usually subordinate it is advisable to operate on not less than 1 gram of the powder, and the J. Lawrence Smith method is to be followed in nearly all its details (p. 144). Because of the presence of a large amount of calcium carbonate in the rock it might seem that addition of precipitated carbonate would be needless. That it is not, however, appears to have been shown by experiments made with and without its addition. A little more alkali was obtained in the former case, a result that is probably due to more effective reaction of the finely divided artificial carbonate than of the crystalline natural carbonate with the ammonium chloride. One-half the usual amount of precipitated carbonate will suffice.

**40. CARBON DIOXIDE, CARBON (WATER).****A. DETERMINATION OF CARBON DIOXIDE.**

This determination is made precisely as under 21. B, page 150. If the absorption tubes have a height of about 10 cm. and a diameter of 1.2 cm. the first one will need refilling after every second analysis. For this reason Liebig or Geissler bulbs containing potash solution may be preferred.

**B. DETERMINATION OF CARBON OF CARBONACEOUS MATTER.**

The character of the carbonaceous matter in limestones has not been made the subject of study, so far as known to me, but it seems reasonable to assume that it resembles coaly matter rather than the indefinite humus. Although on solution of the rock in dilute acids

the odor of the escaping gas is sometimes indicative of volatile organic substances, the amount thus escaping can never be more than a small fraction of the total. If the rocks are boiled with acid the filtrate may now and then be somewhat colored, but if care is taken to use dilute acid and to filter as soon as its action on the carbonates is over, little or no organic matter is likely to pass into solution. Hence, in all such cases the following method for the determination of its carbon is indicated, provided sulphides are practically absent.

Treat 1 to 10 grams of the rock with dilute hydrochloric acid till the carbonate constituents are surely decomposed, filter through an asbestos plug filling a constriction in a glass combustion tube, wash thoroughly with water, dry, and burn the carbonaceous matter over an ordinary burner in an air or oxygen current, collecting the escaping water in a calcium-chloride tube and the carbon dioxide in one filled with soda-lime. The combustion need occupy but a few minutes, as a rule.

If the rock is free from hydrous minerals, if the asbestos likewise yields no water on ignition, and if the asbestos and its load have been thoroughly dried, from the gain in weight of the calcium-chloride tube the hydrogen of the organic matter may be calculated. These conditions will, however, seldom be fulfilled, so that only the carbon has to be considered. In fact, it is quite useless, ordinarily, to attempt to determine the organic matter as such, either directly or by calculation. One must perforce be content with knowing its contained carbon.

#### C. SIMULTANEOUS DETERMINATION OF WATER AND OF TOTAL CARBON IN BOTH FORMS.

In presence of sulphides or soluble organic matter the following method will sometimes answer for the simultaneous determination of the first two or even all three of the above enumerated constituents.

Ignite in a current of air 1 gram of the rock in a tube of very hard glass, or in the tubulated crucible of Gooch (fig. 15, p. 63), collecting the water and the carbon dioxide as in B. The sulphur of sulphides will be retained as calcium sulphate. It is necessary for the success of this operation that the temperature be sufficient to drive out all carbon dioxide from the carbonates, but insufficient to decompose calcium sulphate. The water found represents that in the minerals of the rock as well as that derived from the hydrogen of the organic matter.

A separate determination of the carbon dioxide (see A., p. 186) gives the data for calculating the carbon of the organic matter.

According to G. T. Morgan<sup>a</sup> the two conditions of carbon can be successively determined on the same portion of sample by first finding

<sup>a</sup> Jour. Chem. Soc., vol. 85, 1904, p. 1001.



the carbon dioxide as in A, but with substitution of orthophosphoric for hydrochloric acid, whereupon, after weighing and replacing the absorption tubes, chromic acid is added and the boiling renewed to oxidize the organic matter.

#### 41. CHLORINE.

Several grams of the rock are dissolved in the cold, or with as low a heat as may be, in dilute nitric acid free from chlorine, the solution is filtered, silver nitrate added, and the precipitate collected and treated as usual. (See 22. A, B, and C, pp. 153-154.)

If the rock is likely to contain chlorine-bearing silicates, it is decomposed with its own weight or less of chlorine-free sodium carbonate in a platinum crucible over the burner or moderate blast without undue prolongation of the heating. The mass is extracted with hot water and the filtrate acidified with cold dilute nitric acid, or it may be decomposed directly with the acid and the solution filtered. The further treatment is as above given.

#### 42. FLUORINE.

As with silicate rocks, an exact determination of fluorine in carbonate rocks is difficult if not impossible with our present methods, and little or nothing has been done along this line.

Because of the great preponderance of lime, direct fusion with sodium-potassium carbonate and silica is of somewhat doubtful practicability. Probably the following treatment will afford the best results.

Dissolve the powder in dilute acetic acid, as far as this may be possible without boiling, and filter. To the filtrate add solution of sodium carbonate till in some slight excess, boil, and treat the precipitate of calcium carbonate and possible fluoride as on page 157. Most of the fluorine that may have been dissolved by the acetic acid will thus be found. The siliceous matter insoluble in acetic acid is fused with sodium-potassium carbonate and further treated as in 23. B. a, page 155.

#### 43. SULPHUR.

##### A. TESTS AS TO ITS CONDITION. DETERMINATION OF SULPHATE SULPHUR.

In very many, perhaps most, carbonate rocks any sulphur present is chiefly if not wholly in the sulphide condition, and then usually as pyrite. In what condition it may be is readily ascertainable. If moistened lead-acetate paper is blackened when held over the mouth of a test tube in which some of the rock powder is being boiled with

dilute hydrochloric acid, a soluble sulphide is indicated, but it is seldom more than a negligible trace. If the filtrate after approximate neutralization with ammonia gives a precipitate with barium chloride there is sulphate present. If the total sulphur obtained as in B, below, is in excess of that in the sulphate and soluble-sulphide states, the difference may safely be calculated to iron disulphide.

In the presence of much sulphide and also of sulphate sulphur the extraction of the latter should be made in an atmosphere of carbon dioxide, but usually this precaution is quite unnecessary.

## B. DETERMINATION OF TOTAL SULPHUR.

### a. METHODS OF CONVERTING SULPHIDES TO SULPHATES.

*α. By ignition without flux.*—In a rock that is not heavily charged with sulphides the following method of my own has afforded excellent results, even in the presence of 1 per cent of organic matter, though its range of application has not been ascertained.

The platinum crucible containing 1 or 2 grams of the rock powder is placed in the perforated disk (fig. 3, p. 30) and exposed to the heat of a burner for fifteen minutes or to that of an inclined and moderate blast for ten minutes. So far as present experience teaches, it is not at all necessary to begin with a gentle heat. All sulphide sulphur is oxidized and retained by the lime as sulphate without any loss by volatilization, all organic matter is removed, and the silicates are rendered soluble in acid if they are not in excess. (See 32. A. b. *α*, p. 175.)

*β. By ignition with sodium carbonate.*—One to 2 grams of powder are mixed with half the weight of sulphur-free sodium carbonate in a platinum crucible and heated in the perforated disk, as in *α*. It is not probable that addition of niter is called for, even when a good deal of carbonaceous matter is present. Since not enough flux is used to produce more than a sintering, the air entering the crucible after the bulk of the carbon dioxide has passed off effects very speedy oxidation in the porous mass.

With highly impure limestones it may be necessary at times to increase the amount of flux and to use a little niter as well.

### b. TREATMENT AFTER IGNITION.

The ignited mass obtained in a, *α* or *β*, will usually separate easily from the crucible. It is transferred to a small beaker and covered with water. The crucible is cleansed with dilute hydrochloric acid and the solution poured into the beaker. More acid is added till decomposition is complete in the cold or on gently warming. The solution is then filtered, diluted if need be to 150 or 200 cm.<sup>3</sup>, brought to boiling or simply placed on the steam bath, barium chloride added, and the precipitated sulphate collected in due time and weighed as usual.

Evaporation to dryness to separate the dissolved silica is quite unnecessary. After decomposition with acid the solution should never be permitted to gelatinize, and it needs but little practice on the part of the operator to insure unfailing success in preventing this. If it should happen, however, it is generally best to begin over, but the difficulty may be overcome if but little free acid is present by dissolving the gelatinized silica in sulphur-free fixed alkali and reacidifying in the cold with hydrochloric acid.

#### 44. WATER.

##### A. HYGROSCOPIC WATER.

From one to several grams of the air-dry powder are heated in a current of air dried by calcium chloride in a glass tube at 100 to 105° and the escaping water collected in a calcium-chloride tube, or the air may be dried by sulphuric acid and the water collected in sulphuric acid. This direct determination of the water is certain to give a higher and more correct result than that obtained by drying in a crucible at the above temperature.

##### B. COMBINED WATER.

This may be ordinarily determined on the air-dry sample by heating in a tube of combustion glass and collecting the water in a calcium-chloride tube with the above-mentioned precautions. Or the powder may be mixed with dry sodium carbonate and heated in the apparatus of Gooch (5. C. b, p. 62) or with anhydrous borax according to Jannasch (5. C. d, p. 66). From the result thus found the separately determined hygroscopic water is to be deducted. It is to be remembered that the result will be in error by the amount of water afforded by the hydrogen of any organic matter there may be in the limestone.

### PART III.—CONDENSED ANALYSIS.

For many purposes, especially those with a technical end in view, a highly refined or detailed analysis is not called for. The analyses of this kind that are now made vastly exceed in number those demanding the kind of work provided for in many of the sections of Part II. To meet the needs of those having work of this kind to do, a procedure is here given in brief, that covers the important constituents of limestones. The operations are generally given without explanations. Those who seek reasons for one or another procedure or details of manipulations are referred to the corresponding sections of Part II.

## 45. DECOMPOSITION AND SOLUTION.

If the substance is convertible by blasting into a condition wholly decomposable by hydrochloric acid, one-half to 1 gram of the fine powder is strongly ignited in a covered platinum crucible over the blast for fifteen minutes, or longer if the blast is not very powerful.

If the material is very impure, one-half to 1 gram is to be mixed with half its weight of pure sodium carbonate and ignited over the burner and blast till well sintered together.

The ignited material obtained in either of the above ways is transferred to an evaporating dish, preferably of platinum, and moistened with a few cubic centimeters of water. The crucible is cleaned with dilute hydrochloric acid, the solution poured into the dish, which is to be kept covered if sodium carbonate was used, and more acid added. Solution of the mass may be aided by gentle pressure with the flattened end of a glass rod. The solution is then evaporated to dryness on the steam bath. It will do no harm with limestones low in magnesium to hasten dehydration of the silica by placing the dish with its dry contents in an air bath at an elevated temperature or on a triangle resting on a hot plate at 200° for an hour or less.

## 46. SILICA.

The residue is treated with 5 to 10 cm.<sup>3</sup> of strong hydrochloric acid, which is then diluted to half strength, or upon the residue may be poured at once a larger volume of acid of half strength. The dish is then covered and digestion allowed to go on for ten minutes on the bath, after which the solution is passed through a 7-cm. paper and the silica washed with water, cold at first and hot later, or with hot dilute hydrochloric acid at first, followed by a little hot water. The filtrate is again evaporated to dryness, this time only on the steam bath, after which the residue is digested with acid as before, but in smaller amount, and the second portion of usually dark-colored silica is collected on another small paper. The filter papers with their contents are slowly charred in a platinum crucible, then ignited, first over the burner, finally over the blast for five to fifteen minutes, according to the amount of the silica. If the blast is weak, a second application of it should follow the first weighing. The finally weighed silica is moistened with water and two drops of sulphuric acid and 5 to 10 cm.<sup>3</sup> of pure hydrofluoric acid are added, the crucible is placed in a radiator (fig. 2, p. 29), and the liquid wholly evaporated. The residue is blasted for a minute and in due time weighed. It is better not to make this correction if it is not intended to separate and determine also the silica always accompanying the iron and aluminum oxides, but it should never be omitted if this latter correction is contemplated.

## 47. ALUMINUM, IRON, ETC.

The filtrate, which need not exceed 100 cm.<sup>3</sup> in bulk with fairly pure limestones, is brought to boiling in a beaker, or preferably in platinum, and made alkaline with ammonia after adding a few cubic centimeters of bromine water and if need be enough hydrochloric acid to insure a total of 10 to 15 cm.<sup>3</sup> of strong acid. The ammonia should not be in such excess as to require long boiling to expel the most of it, nor is the expulsion of the whole of it necessary or indeed desirable. As soon as the precipitate settles the solution is passed through a filter of suitable size and the precipitate is washed a few times with hot water and sucked fairly dry. It is redissolved in hot hydrochloric acid and precipitated again by ammonia in presence of bromine, but with less ammonium chloride and in a smaller bulk of solution. If the filter is still in good condition, it is used also for this precipitate, which is washed with hot water and sucked dry at the pump. With high iron and magnesium the precipitation may well be repeated.

The re-solution of the first precipitate may be made with nitric acid if thought desirable. Its use involves very little washing of the second precipitate. To reduce the amount of aluminum passing into the filtrate, the precipitated oxides may be washed with water containing in the liter 20 cm.<sup>3</sup> of nitric acid that has been neutralized with ammonia.

The paper containing the final precipitate is placed in a platinum crucible, slowly charred, then ignited over a gradually increased flame, and finally over the blast for two to five minutes. The weight found represents the oxides of ferric iron, aluminum, titanium, phosphorus, and manganese, the last presumably  $Mn_2O_4$ .

The iron, titanium, and silica in the ignited oxides are determined, after fusion with potassium or sodium pyrosulphate, exactly as detailed in 33.C, p. 180. The phosphorus and manganese, having been determined in different portions (37, p. 184, and 34. B, p. 182), the aluminum oxide is found by subtracting all these oxides from the original weight of the mixture. Often the percentage reported as alumina includes in reality the titanium, phosphorus, and some of the manganese, where no attempt has been made to separately determine these.

## 48. CALCIUM.

The combined filtrates from the iron, aluminum, etc., are concentrated if need be to a bulk of 200 to 250 cm.<sup>3</sup>, with occasional addition of a few drops of ammonia. If a slight precipitate separates, it is collected on a small filter, washed with water, redissolved in hydrochloric acid, and reprecipitated by ammonia. It is added to

and treated with the main quantity of iron and aluminum oxides, and the filtrates are combined. To them is added while boiling 20 cm.<sup>3</sup> of a saturated solution of ammonium oxalate, and the boiling is continued until the precipitated calcium oxalate assumes a well-defined granular form. It is then allowed to stand for an hour, filtered, and washed a few times with hot water. The precipitate and filter are ignited in platinum till the oxalate is converted to oxide, which is redissolved in hydrochloric acid and the solution made up to 100 cm.<sup>3</sup>. Ammonia is added in slight excess to the boiling solution and also ammonium oxalate to reprecipitate the lime. After an hour the oxalate is collected, washed a few times with small amounts of hot water, ignited over the blast, and weighed as the oxide.

Instead of igniting and weighing as oxide it is permissible to dissolve the oxalate in sulphuric acid, taking care to extract the filter thoroughly with hot dilute acid, and to titrate the solution with permanganate.

## 49. MAGNESIUM.

To the combined filtrates from the calcium precipitations are added sodium-ammonium phosphate solution in excess and, when the precipitate has formed after vigorous stirring, ammonia in considerable excess. After several hours, twelve or fifteen if the precipitate is very slight, the solution is filtered, the precipitate redissolved in hydrochloric acid and reprecipitated by ammonia in moderate excess and a drop of the phosphate precipitant in the usual manner. After two hours the precipitate is filtered on paper or on a Gooch filter, ignited, and weighed as pyrophosphate.

## 50. ALKALIES.

See 39, p. 186.

## 51. CARBON DIOXIDE.

See 40, p. 186.

## 52. SULPHUR.

See 43, p. 188.

## 53. WATER.

See 44, p. 190.

## 54. IGNITION LOSS.

This determination has little significance, though it affords a more or less correct indication of the proportion of carbonates in the rock. Since it is a determination commonly made in commercial laboratories, some space will be devoted to it.

Loss on ignition with carbonate rocks represents, as with those of a silicate nature, the algebraic sum of a number of chemical changes involving both losses and gains, and its amount will depend largely on the temperature employed. At the temperature of a moderate

blast, with crucible covered, carbon dioxide, water, and carbonaceous matter escape wholly. Sulphides are oxidized to sulphates and all the sulphur is retained as sulphate by the calcium. With a powerful blast the sulphate is decomposed gradually, with eventual loss of the entire sulphur. At this point the alkalies begin to escape as oxides, the potassium relatively faster than the sodium, and they may be found in part condensed on the lid of the crucible. If the heating is long continued they can be wholly volatilized. As a small offset to these losses the iron of pyrite and the iron and manganese of ferrous and manganous carbonates take up oxygen and unite with the lime. By proper regulation of the temperature it is not difficult to prevent loss of sulphur and alkalies, and this is the object to be sought in making the determination.

One-half to 1 gram of the powder is placed in a platinum crucible of 20 to 25 grams weight, the crucible is inserted to three-fifths of its depth in a perforated platinum disk or asbestos board (fig. 3, p. 30), and an inclined blast flame of considerable power is caused to play against the bottom of the covered crucible for fifteen or twenty minutes. A repetition of the heating for five minutes will usually cause little or no further loss. Duplicate determinations should agree within 0.2 per cent, and after the operator becomes skilled probably within 0.1 per cent. A muffle can be substituted for the blast. In my experiments the crucibles occupied a position 2 inches inside the closed door of the muffle. It is, however, important that each operator should ascertain just what the proper conditions may be for his particular blast or muffle. They are right if after the ignition he finds exactly the whole of the sulphur in the ignited powder in the sulphate state.

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[Bulletin No. 305.]

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THE DIRECTOR,

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JANUARY, 1907.

( )











Bulletin No. 306

Series { B, Descriptive Geology, 109  
F, Geography, 54

DEPARTMENT OF THE INTERIOR  
UNITED STATES GEOLOGICAL SURVEY

CHARLES D. WALCOTT, DIRECTOR

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# RATE OF RECESSION OF NIAGARA FALLS

BY

G. K. GILBERT

ACCOMPANIED BY A REPORT ON THE SURVEY OF THE CREST

BY

W. CARVEL HALL.



WASHINGTON  
GOVERNMENT PRINTING OFFICE  
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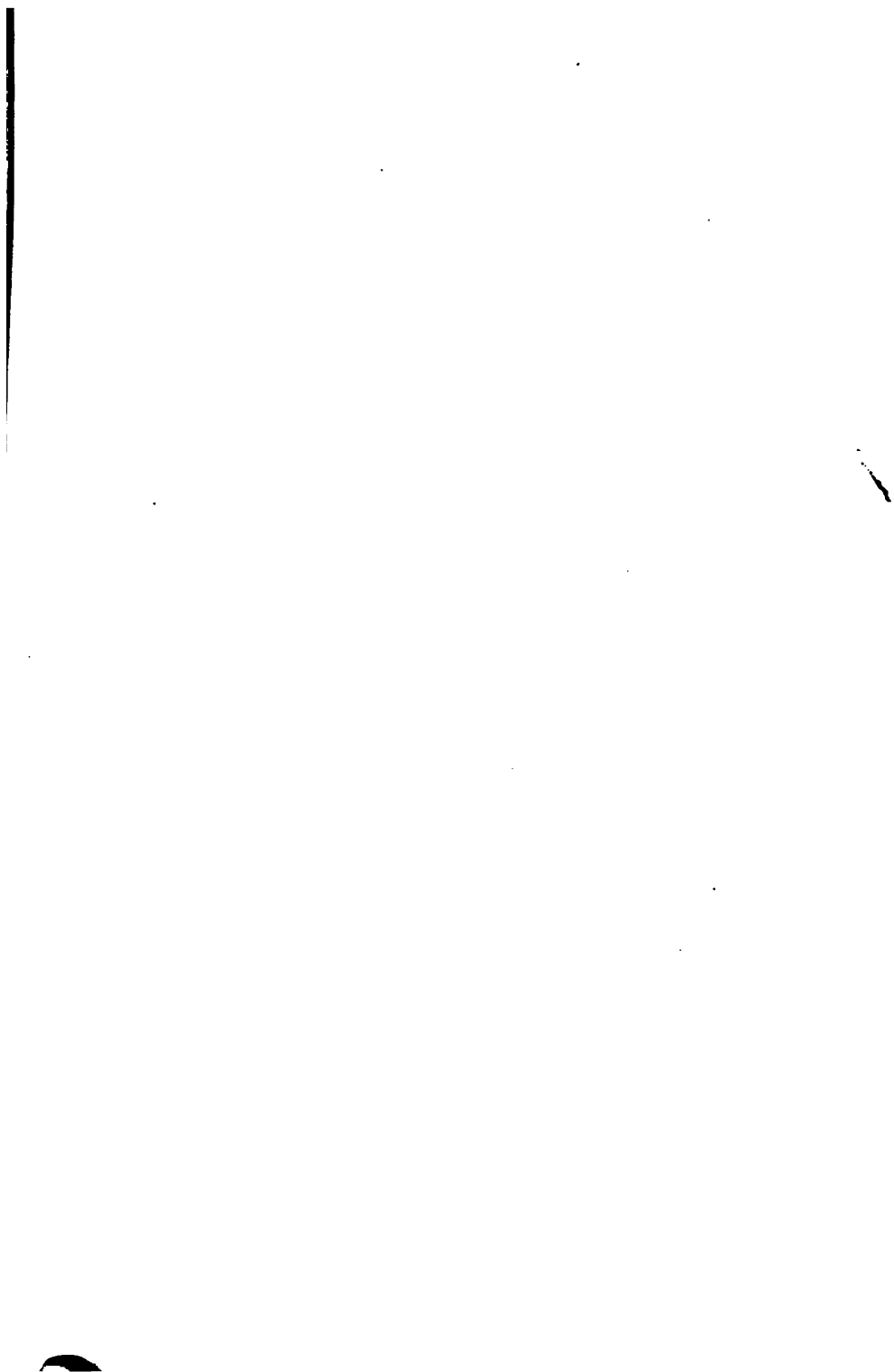
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1





### THE HORSESHOE

Dated and printed July 4, 1886. The head of the Horseshoe is now *more* rapidly than any other part of the cataract. The notch in its further margin was developed after 1827.

# RATE OF RECESSION OF NIAGARA FALLS.

By G. K. GILBERT.

## INTRODUCTION.

The erosive work of the cataract of Niagara is exceptionally rapid. This depends primarily on the great power of the falling water, but in part on the character of the local geologic structure. The rocks are stratified and lie nearly level. The upper layers are of limestone, strong and resistant; the lower, consisting chiefly of shale, are comparatively weak and yielding. As the shales are worn away below the limestone beds are undermined, so that their edges project like a cornice and are deprived of support. From time to time they yield to the force of their own weight and fall away in large blocks. Each rock fall causes a jar of the ground which is perceived by people in the vicinity, and results in a modification of the crest of the cataract which is readily seen by anyone familiar with its outline. Such changes of the crest have been observed from time to time ever since the neighboring banks of the river were occupied by white men. It is highly probable that they were also observed by Indians before the advent of white men, but on this point I have made no inquiries, as Indian traditions are not likely to be sufficiently definite to aid in determining the rate of progressive change in the position of the cataract.

The surface of Lake Erie is 325 feet higher than the surface of Lake Ontario. The belt of land between them includes two plains, of which the higher and broader is raised but little above the level of Lake Erie, and the lower slopes gently to the shore of Lake Ontario. The descent from the upper to the lower is abrupt, constituting a line of cliffs parallel to the shore of Ontario and known as the Niagara

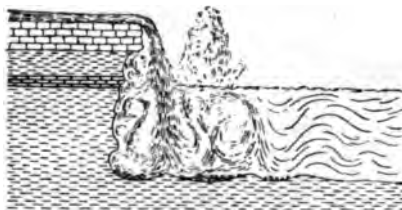


FIG. 1.—Diagrammatic profile of Horseshoe Fall, illustrating mode of erosion and recession.

escarpment. The river, issuing from Lake Erie at Buffalo, flows at first on the upper plain. It is there broad and comparatively shallow and has no valley. At the falls it suddenly drops into the head of a narrow gorge which is 6 miles long and extends to the escarpment. Within the gorge it is narrow and contained by steep walls. Near the head of the gorge the water is deep, the current moderate, and the descent small, but farther on are fierce rapids with steep descent. Some of these relations are shown in fig. 2. As the falls

are at the head of the gorge, it is evident that their recession makes the gorge longer.

Among the early observers of the falls was McCauslin, who remained there from 1774 to 1783. After describing the escarpment at Queenston he says:<sup>a</sup>

It is universally believed that the cataract was originally at this ridge, and that it has by degrees worn away and broke down the rock for the space of these six or seven miles. Some have supposed that from these appearances, conjectures might be formed of the age of this part of the world. To enter upon such a calculation, it would previously be necessary to ascertain how much the fall had retired in a hundred years, or any other certain period. Suppose that we were even in possession of such a fact, still the conclusions drawn from it would be liable to the greatest uncertainty, as it is evident that the space of rock broke down and worn away in a certain number of years would not always be the same. The more or less hardness and brittleness of the rock in different



FIG. 2.—Bird's-eye view of Niagara River. The view is southward, or upstream, from a point above the shore of Lake Ontario, and shows the two plains, the escarpment, and the gorge. B, Buffalo. NF, Niagara Falls. L, Lewiston. Q, Queenston. EE, Niagara escarpment.

parts; the greater or less severity of the frosts in different years; and the quantities of water that flowed at different periods in the cataract of the river, would all occasion considerable variations. This retrocession of the Falls does not by any means go on so quickly as some have imagined. During nine years that I have remained at Niagara, very few pieces of the rock have fallen down which were large enough to make any sensible alteration in the brink; and in the space of two years I could not perceive, by a pretty accurate measurement, that the North-East brink had in the least receded. If we adopt the opinion of the Falls having retired six miles, and if we suppose the world to be 5,700 years old, this will give about sixty-six inches and an half for a year, or sixteen yards and two thirds for nine years, which I can venture to say has not been the case since 1774.

<sup>a</sup> McCauslin, Robert, An account of an earthy substance found near the Falls of Niagara and vulgarly called the spray of the falls, together with some remarks on the falls: Trans. Am. Philos. Soc., vol. 3, 1793. (Read October 16, 1789.) The passage cited is on pages 23-24.

Enys, who visited the cataract in 1787, quotes the opinion of residents that "the Falls have altered their position or retreated since the memory of men,"<sup>a</sup> but dissents from the view (which seems also to be generally entertained) that the original situation of the falls was at Queenston.

Weld, whose visit to the falls was in 1796, says that "even within the memory of many of the present inhabitants of the country, the falls have receded several yards."<sup>b</sup>

He favors the theory that the gorge from Lewiston to the falls was made by the falls, and his discussion of the subject shows him to have been a close observer and clear thinker.

Volney two years later repeats the general statement of observed recession, and adds:<sup>c</sup>

If the European colonists or travellers, to whom this region has been accessible for a century and a half, had made careful memorandums, from time to time, of the state of the fall, we should, by this time, have been able to trace the progress of those revolutions, which are easily proved to have taken place, by vestiges and indications which present themselves at every step.

And still further, in a footnote:<sup>d</sup>

It is extremely desirable that the government of the United States, at present under the direction of a friend to the arts and sciences [Jefferson], should order to be drawn up an exact description of the present state of the cataract. This statement, compared with subsequent appearances, observed from time to time, would enable us to trace with certainty the changes that may hereafter take place.

Francis Hall, 1816, says:<sup>e</sup>

The name of "the Horse shoe," hitherto given to the larger Fall, is no longer applicable: it has become an acute angle. \* \* \* An officer who had been stationed in the neighbourhood thirty years, pointed out to me the alteration which had taken place in the centre of the Fall, which he estimated at about eighteen feet in the thirty years.

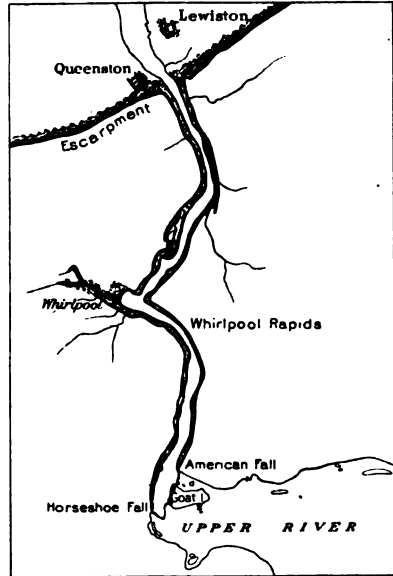


FIG. 3.—Map of the Niagara gorge, showing its relations to the falls and the escarpment.

<sup>a</sup> Rept. Canadian Archives, 1886 (published 1887), p. ccxxxii.

<sup>b</sup> Weld, Isaac, jr., *Travels through the States of North America and the Provinces of Upper and Lower Canada during the years 1795, 1796, and 1797*, London, 1799, p. 320.

<sup>c</sup> Volney, C. F., *A view of the soil and climate of the United States of America, etc.*, translated by C. B. Brown, Philadelphia, 1804, p. 92.

<sup>d</sup> *Op. cit.*

<sup>e</sup> Hall, Lieut. Francis, *Travels in Canada, and the United States, in 1816 and 1817*, Boston; republished from the London edition by Wells & Lilly, 1818, p. 144.

Gilpin, whose visit was probably a few years later, says:<sup>a</sup>

The toe of the shoe, however, is now an angle, rather than a curve; but the inhabitants and early visitors affirm that it was formerly more round, and has gradually assumed its present angular form, within their recollection. \* \* \* Mr. Forsyth, who has resided upon the spot for more than forty years, says, that within his recollection, the centre of this fall has receded from ten to fifteen yards; and, as some intelligent travellers have placed upright a few large stones in front of the hotel, which, when taken in a line, point exactly to that spot, it will of course be ascertained, at the end of a certain number of years, how much this centre recedes annually.

Schoolcraft, whose visit was in 1820, describes the undermining of the limestone by the fretting away of the shale, and mentions with approval the theory that the falls were anciently at Lewiston. He says:<sup>b</sup>

\* \* \* The wasting effects of the water, and the yielding nature of the rocks, remain the same, and manifest the slow process of a change, at the present period, as to position, height, form, division of column and other characters, which form the outlines of the great scene; and this change is probably sufficiently rapid in its operation, if minute observations were taken, to imprint a different character upon the Falls, at the close of every century. Nothing in the examination of the geological constitution, and mineral strata of our continent, conveys a more striking illustration of its remote antiquity, (still doubted by many) than a consideration of the time, it must have required for the waters of Niagara, to have worn their channel, for such an immense distance, through the rock. It is true, we are in possession of no certain data, for estimating the annual rate of their progress, or for comparing the results with the Mosaic history of the earth. All that can be presumed is, that this progress, is now as rapid, as it was in former ages. \* \* \*

Maps and descriptions are now extant, which will enable us to fix the rate of its progress, on the expiration of the present century, and we should not be disappointed in our anticipations, if its progress is found, greatly to exceed the prevalent expectation. To aid in the determination, the Island of Iris, which extends from the brink of the Fall, up the river, and which is now connected with the shore, by a wooden bridge, appears to present great facilities. A simple measurement of its length, with a monument for recording it at its head, would convert it into a graduated scale, and the point of the indentation of the Horse Shoe Fall, could, in like manner, be perpetuated on either shore, by a series of corresponding celestial observations, for determining the longitude of the extreme point of that incurvation. Distant ages would thus be furnished with data, the precision of which, would probably enable them to throw new and important lights on the history of the earth, and the changes it has undergone. Is this suggestion of too visionary a nature, to merit the consideration of geological societies?

Capt. Basil Hall, R. M., made a study of the cataract in 1827, and two years later published an excellent description, from which I quote:<sup>c</sup>

In the course of our investigations and rambles, we met a gentleman who had resided for the last thirty-six years in this neighborhood—happy mortal! He told us that the Great Horse Shoe Fall had, within his memory, gone back forty or fifty yards—that is to say, the edge, or arch of the rock over which the water poured, had broken down from time to time to that extent. This account was corroborated by that of another gentleman, who had been resident on the spot for forty years.

<sup>a</sup> Gilpin, H. D. A northern tour; being a guide to Saratoga, Lake George, Niagara, Canada, Boston, etc., Philadelphia, 1825, p. 149.

<sup>b</sup> Schoolcraft, Henry R., Narrative journal of travels, etc., made in 1820, Albany, 1821, pp. 45-47.

<sup>c</sup> Hall, Capt. Basil, Travels in North America in the years 1827 and 1828, vol. 1, Edinburgh, 1829, pp. 195-197.

As these statements came from persons of good authority, I was led to examine the geological circumstances more minutely; for I could not conceive it possible, that the mere wearing of the water could perform such rapid changes upon hard lime-stone. The explanation is very simple, however, when the nature of the different strata is attended to. In the first place, they are laid exactly horizontal, the top stratum being a compact calcareous rock. In the next place, I observed, that in proportion as the examination is carried downwards, the strata are found to be less and less indurated, till, at the distance of a hundred feet from the topmost stratum, the rock turns to a sort of loose shale, which crumbles to pieces under the touch; and is rapidly worn away by the action of the violent blasts of wind, rising out of the pool into which this enormous cascade is projected.

In process of time, as the lower strata are fairly eaten or worn away, the upper part of the rock must be left without a foundation. But owing to the tough nature of the upper strata, they continue to project a long way over before they break down. There must come periods, however, every now and then, when the overhanging rock, with such an immense load of water on its shoulders, will give way, and the crest, or edge, of the Fall will recede a certain distance. At the time of our visit, the top of the rock, or that over which the river was directed, overhung the base, according to the rough estimate I made, between 35 and 40 feet, thus forming a hollow space, or cave, between the falling water and the face of the rock.

While the above lines were actually in the printer's hands, my eye was accidentally caught by the following paragraph in a newspaper:—

"**NIAGARA FALLS.**—A letter from a gentleman at that place, dated Dec. 30, 1828, states, that on the Sunday evening preceding, about 9 o'clock, two or three successive shocks or concussions were felt, the second of which was accompanied by an unusual rushing sound of the waters. The next morning it was discovered, that a large portion of the rock in the bed of the river, at the distance of about two-fifths from the Canada shore to the extreme angle of the Horse Shoe, had broken off, and fallen into the abyss below. The whole aspect of the Falls is said to be much changed by this convulsion. A course of high winds for several days previous to its occurrence, producing an accumulation of water in the river, is supposed to have been the immediate cause. This gradual crumbling away of the rock over which the Niagara is precipitated, adds plausibility to the conjecture, that the Falls were once as low down as Lewistown, and have for centuries been travelling up towards their present position."

Captain Hall also published a series of sketches of the falls,<sup>a</sup> and as these were made with the camera lucida they have exceptional value. They, in fact, constitute the first record bearing on the rate of recession from which measurements can profitably be made, and there is frequent reference to them in other parts of this paper.

The preceding citations serve to show the early development of three ideas: (1) That the crest of the Horseshoe Fall is receding upstream, the recession being caused by the energy of the cataract; (2) that the gorge below the falls was created by this process of recession, the position of the falls having originally been where the mouth of the gorge now is, and (3) that it is possible, by sufficiently accurate observations, to determine the rate at which the change is taking place.

Associated with the idea of measuring the rate of recession was that of applying it to the determination of the time consumed by the river

<sup>a</sup> Hall, Capt. Basil, Forty etchings, from sketches made with the camera lucida, in North America, in 1827 and 1828, Cadell and Co., Edinburgh, 1829, pls. 1-5.



in the making of the gorge. By some of the earlier writers the age of the gorge was obscurely connected with the age of the world as estimated from Biblical data; by others it was recognized as a small fraction of geologic time. With the progress of knowledge of the local geologic history there was increasing interest in the time estimates for the river, and the various conditions affecting the estimate came to be scrutinized with much care. As developed by careful study, the problem proved to be complex and difficult. It came to be recognized not only that the rate of recession in different parts of the gorge must have varied with the height of the cataract, the temporary width of the stream, and the thickness of the capping limestone, which is different in different places, but also in a very important way with the volume of water carried by the river, which has been subject to extreme fluctuations. The influence of these various conditions assumed prominence in the discussion, and altho the rate of present recession came to be fairly well known, opinions still differed widely as to the total period represented by the gorge. The age of the gorge is outside the scope of the present paper, and the subject is here mentioned only to show the basis of the strong interest which has been felt in the determination of the present rate of recession.

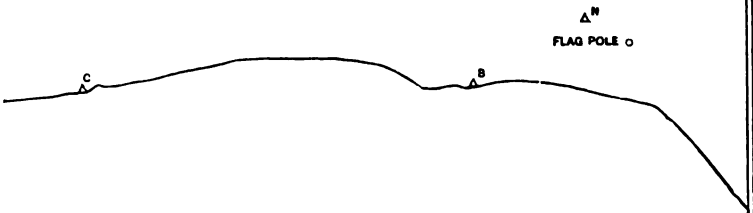
In 1841 James Hall, then geologist of the fourth district of New York, undertook the preparation of an authoritative map of the crest of the falls, and employed for that purpose E. L. Blackwell, a civil engineer. The work was completed in the autumn of 1842, at which time a series of monuments were established at the principal trigonometric points. The map was published the following year,<sup>a</sup> together with descriptions of the monuments and a table of compass bearings from the various trigonometric points to objects whose positions were determined by the method of intersection. It was the purpose of this survey to make definite record of the existing position of the crest line and connect this record with permanent monuments, so that by means of a similar survey at some future time the extent of changes might be determined. This purpose it has served. Monuments then placed have been used as starting points in subsequent surveys, and two of them are still extant.

As this work by our great master in geology marks a turning point in the subject—the change from the vague to the definite—I quote a few passages to show his point of view:

Among the phenomena of waterfalls and river gorges, the Cataract of Niagara is justly regarded as holding the first rank, and as standing an index in the path of time, by which the influence of numberless ages upon the surface of our planet may be recorded. Its present, its former and its prospective conditions have engaged the investigation and speculation of many philosophers. The possible consequences of its entire reduction, and the drainage of the upper lakes, have excited the wonder and the apprehensions of many. The

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<sup>a</sup> Nat. Hist. New York, pt. 4. Geology, 1843, opp. p. 102.



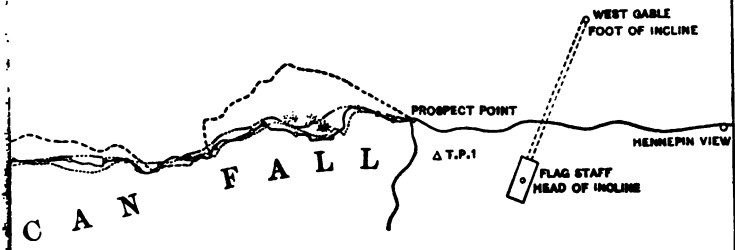
DEPARTMENT OF THE INTERIOR  
UNITED STATES GEOLOGICAL SURVEY  
CHARLES D. WALCOTT, Director

# LINE OF NIAGARA FALLS

SURVEYS MADE IN 1842, 1875, 1886, 1890, and 1905  
COMPILED BY  
CARVEL HALL, TOPOGRAPHER U. S. G. S.



- EXPLANATION
- Survey of 1842
  - Survey of 1875
  - Survey of 1886
  - Survey of 1890
  - Survey of 1905





estimated time of its recession has sprinkled grey hairs among the fresh locks of the young and blooming earth, and alarmed those who would consider her still youthful in years.

But amid all these speculations, Niagara still remains; the thunder of its cataract still reverberates through its deep chasms, and its ocean of waters still rolls on as, unknown to the white man, it rolled a thousand years ago. When we come to the investigation of facts, we find that, except to travellers and the aborigines, Niagara was unknown until within the last fifty years; and that even during this time no accurate observations have been made, no monument erected to determine whether the falls are retrograding or not. The testimony of living witnesses and historical evidence unite in confirming the opinion that the water is wearing away the rock, and that the outline of the falls has changed. From these general observations, it has been estimated that they have receded at the rate of about forty feet in fifty years. Without pretending to question the accuracy of this or any other estimate of the kind, or to establish any rate of retrogression in the falls, we may examine its present, and from numerous facts infer its past condition; and from these we are entitled to draw an inference for the future, though without specifying time.<sup>a</sup>

The foresight with which he planned the survey and record for the specific purpose is shown by the statement with which the map is introduced:

The accompanying map has been constructed from a very careful survey by Mr. Blackwell, giving the present position and outline of both falls, and the river banks upon either side. Upon application to His Excellency Sir Charles Bagot, late Governor-General of Canada, I was authorized to establish monuments upon the Canada shore, and was also kindly offered every other aid to promote the objects of the survey. These monuments, together with those in New-York, will enable future observers to ascertain the amount of recession during any given period. In places where the rock is exposed, copper bolts have been fixed, and in other places hewn stone monuments. The starting point for all these observations is a copper bolt fixed in the rock on the north side, near the edge of the American fall. \* \* \* <sup>b</sup>

In 1875 the second survey of the crest line was made by the United States Lake Survey under the direction of Maj. C. B. Comstock, the field work being by F. M. Towar. The United States Geological Survey undertook the third survey, which was made by Robert S. Woodward in 1886. The fourth survey was made in 1890, by A. S. Kibbe, under the direction of John Bogart, State engineer of New York, and a very full report was published. In this report the maps of the three preceding surveys are republished, and the crest lines given by those surveys are also placed on the new map.<sup>c</sup> The fifth survey was made in the spring of 1905, by the United States Geological Survey and the State engineer of New York, the work being done by W. Carvel Hall, and his report follows this paper.

The crest lines determined by the five successive surveys are platted together on Pl. II, and their examination demonstrates clearly the gradual retreat of the crest of the Horseshoe Fall. Each mapped crest line is, on the whole, farther upstream than its predecessor, and

<sup>a</sup> Nat. Hist. New York, pt. 4. Geology. 1843, p. 383.

<sup>b</sup> Op. cit., p. 402.

<sup>c</sup> Seventh Ann. Rept. Comrs. State Res. Niagara for fiscal year October 1, 1889, to September 30, 1890, Albany, 1891.

their interspaces are roughly comparable with the time intervals between the making of the surveys; but each of these statements requires qualification. The region of maximum retreat has shifted from one part to another of the crest during the period of observation, so that in any one part the rate of retreat has been irregular; and when the chart is closely scrutinized it is found that the different lines overlap one another at various points, so that if all of them were rigidly accurate their record would show that the crest line had in places advanced downstream, instead of retreating. In the report of the last survey it is suggested that some of these discrepancies may be explained by an actual sliding forward of upper layers of limestone before they toppled over the brink, but the greater discrepancies can not be explained in this way, and the discrepancies as a whole are unquestionably due to errors in the topographic work, chiefly thru failure to identify points previously sighted when intersecting bearings were taken. Fortunately, they are not of such character or extent as to impair the general conclusions to be drawn from the work; but they serve to caution the student against any overrefinement in the discussion of results.

The Erie Canal is supplied with water from the Niagara River at Buffalo, the Welland Canal is supplied from Lake Erie, and the Chicago Drainage Canal draws water from Lake Michigan. All the water thus diverted is withdrawn from the cataract. So also is water diverted from the river above the falls for factory purposes and for use in the generation of electricity. In recent years the diversion for electric power has rapidly increased, and existing charters authorize so large a draft upon the river that it has come to be recognized that the scenic value of the cataract is in peril. A vigorous protest has been made by lovers of natural beauty, and negotiations are in progress for an international agreement to check and regulate the economic exploitation of the river. Whatever the outcome of these negotiations, there is no reason to expect that the natural flow of the river will be restored, and it is believed that from this time onward the natural conditions will be so far interfered with as to modify the rate of recession. As the geologist is primarily interested in the natural rate of recession, the present time is opportune for a summing up of the data. In fact, the survey of 1905 was ordered in view of the change of conditions from natural to artificial.<sup>a</sup>

<sup>a</sup> Since this paper was written it has come to my knowledge that a resurvey of the Niagara River is being made by the United States Lake Survey, the field work for the crest of the falls having been done in the summer of 1906. This will afford an additional datum on the rate of recession, but is not likely to affect the computation to a material extent. The addition of one year to the period of observation will probably be offset by changes occurring within that year. Inspecting the Horseshoe curve in August, 1906, I was confident that a salient near the angle of the curve, which was recorded by the surveys of 1890 and 1905, did not then exist.—G. K. G.

## THE HORSESHOE FALL.

The Horseshoe Fall is at the head of the gorge. From its edges the walls of the gorge run northeastward approximately parallel. The American Fall is at the side of the gorge, 2,500 feet from its head, and is separated from the Horseshoe Fall by Goat Island. A few hundred years ago the two falls were together, the position of the united cataract being somewhere in the neighborhood of the present American Fall. The subsequent retreat of the Horseshoe Fall has had the effect of lengthening the gorge, but the American Fall has not in the same time made an alcove in the side of the gorge. With reference therefore to the question of the age of the gorge, it is the Horseshoe Fall whose rate of recession is important.

The chief data for the estimation of the rate of recession are the maps of 1842 and 1905, the time interval being sixty-three years. The outlines from those maps are shown in fig. 4. These data, like other statistical data, can be discussed in a variety of ways and made to yield widely divergent results—a fact sufficiently illustrated by earlier estimates of the rate of recession based on comparisons of the map of 1842 with that of 1875, 1886, or 1890. The following paragraphs therefore set forth somewhat fully the methods here used, with the principal considerations on which they are based.

In the lengthening of the gorge the river does its principal work in that part of the Horseshoe curve where the current is deepest. The agitation of the plunging water is there so powerful as to roll about the fallen blocks of limestone, using them as tools to grind the shale, and at the same time breaking them up and eventually washing them downstream. The scour maintains a deep hollow beneath this part of the fall, a hollow whose depth is greater than the height of the fall. (Fig. 1, p. 5.) At the sides of the channel, especially near the right bank, where the sheet of falling water is comparatively thin, the fallen blocks are not cleared away, but cumber the base of the cliff. (Pl. X, p. 22.) As the cataract retreats it leaves behind it a deep channel, or elongated pool, in which the current is slow. Below the cataract the gorge is widened at top by the falling away of its banks. When the shale is exposed to the air it becomes subject to frost action, and for a time the limestone ledge above continues to be undermined, but a practical limit is reached as soon as the talus of fallen material covers the slopes of shale, and thereafter the change is exceedingly slow. The real lengthening of the gorge is along that portion of the Horseshoe where the sheet of falling water is heavy enough to clear away the débris and maintain a deep pool. The retreat of the cliff on either side of this portion is secondary, and appears to have little or no bearing on the question of the rate at which the gorge is growing longer. I have therefore restricted attention to the central part of the Horseshoe curve.

As the two crest lines compared are irregular in outline, a certain confusion arises if the recession of different parts is considered separately. At one place the recession seems to have one direction, at another place to have another direction, and various complications ensue when attempt is made to combine measurements made in different directions. In view of this difficulty it has appeared to me both convenient and legitimate to assume some one direction as the general direction of recession and at all points measure the amount of recession on lines parallel to that direction. From an inspection of the crest lines as wholes and in their relation to each other I have inferred such a general direction of recession, and assuming it to apply to the entire central tract of the Horseshoe, have drawn the system of parallel lines seen in fig. 4. There are

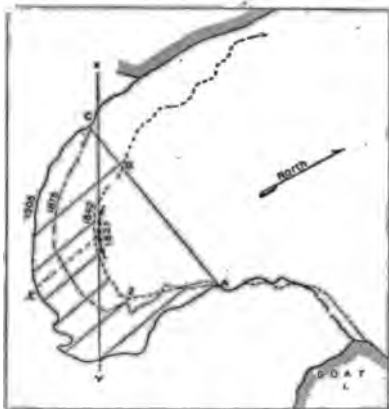


FIG. 4.—Outlines of Horseshoe Fall in 1842, 1875, and 1905, with lines used in computing the rate of recession. The line of crosses suggests a position of part of the crest in 1827.

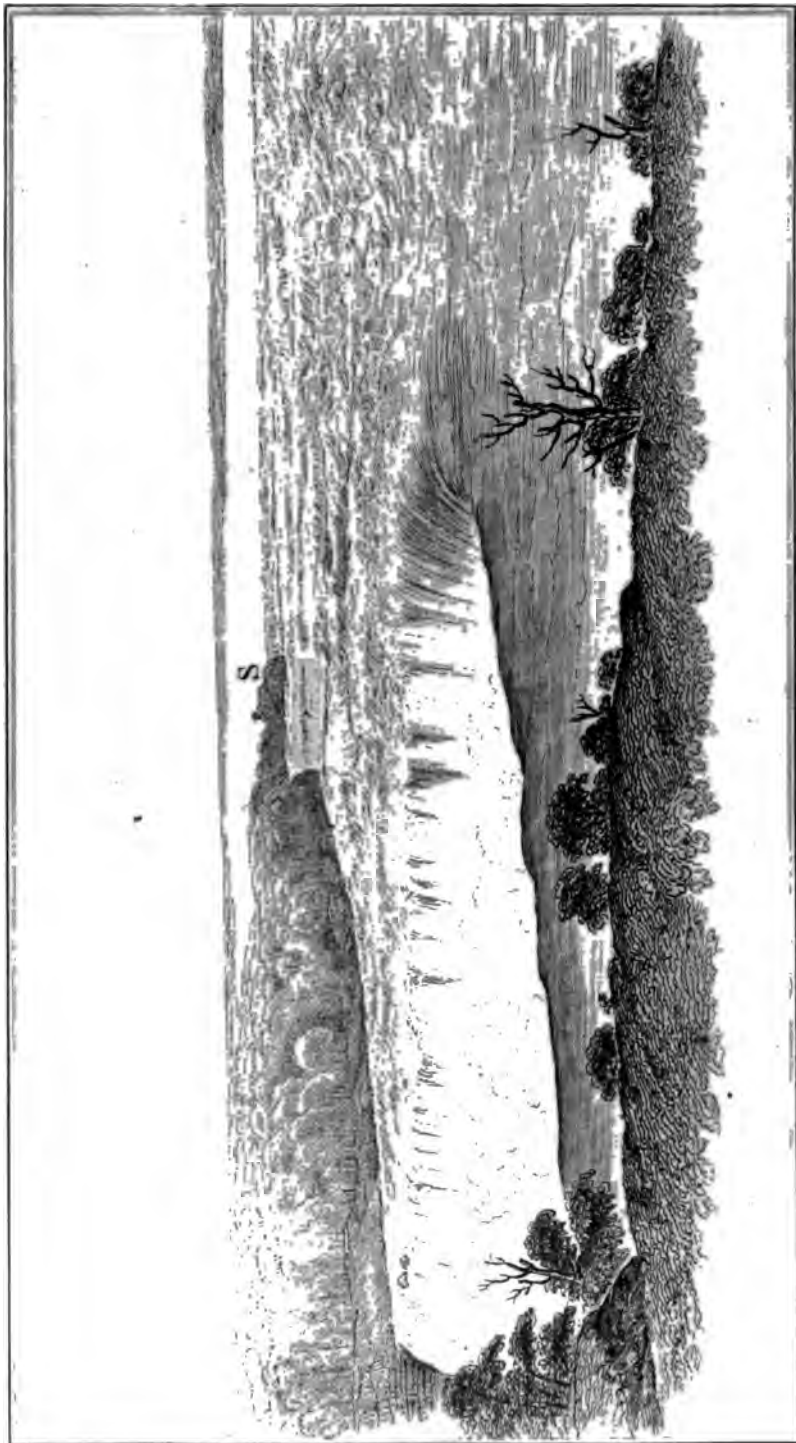
six of these lines, each extending from the crest line of 1842 to that of 1905. Their interspaces, according to the scale of the map, are 100 feet wide. The average length of these lines represents approximately the average recession of the cataract in the part where the sheet of falling water is heaviest. Their lengths are, severally, 430, 292, 260, 276, 317, and 412 feet, giving an average length of 331 feet. This distance divided by the number of years, 63, gives as the average annual recession 5.3 feet.

A somewhat allied method of estimate is concerned with areas.

Still restricting attention to the central portion of the Horseshoe curve, I have drawn a line from A, the point at which the two crest lines begin to diverge, to the opposite shore at C, making its direction lie at right angles to the general direction of recession. The area contained between the two crest lines AZB and AEC, and limited downstream by the straight line AC, may be regarded as the area removed by the central portion of the fall between 1842 and 1905. The corresponding width of this part of the gorge in 1842 was AB, 570 feet; in 1905 AC, 760 feet. The mean of these, 665 feet, is assumed as the average width for the intervening period. The indicated area between the crest lines was found by measurement to be 223,000 square feet, and this quantity being divided by 665 feet, gives 335 feet as the average recession in a direction at right angles to AC. Dividing, as before, by 63, the number of years, I obtain again as an estimate of the average annual rate 5.3 feet.







HORSESHOE FALL IN 1827.

Copy of sketch by Capt. Basil Hall, made with camera lucida, from veranda of Forsyth's Hotel.



HORSESHOE FALL IN 1895.

Photograph from same point as sketch, Pl. III. The island at S is common to the two views. Their comparison shows the recession of the fall and the change in its outline.



The close coincidence of these two results is accidental, altho a general agreement was to be expected because the assumptions underlying the computations are harmonious. As already stated, materially different results may be obtained with different assumptions.

Less harmonious results are obtained if the period from 1842 to 1905 is divided into parts and the parts are separately computed. Their discordance has two sources which can not be fully discriminated. From the nature of the case the rate of recession is not uniform. The distance to which the cornice of limestone comes to project before it is broken away depends not only on the strength of the rock, but on the local arrangement of vertical joints by which it is traversed, and also to some extent on the shape of the temporary outline of the crest. The fall of rock is therefore irregular and only obscurely rhythmic. In a period measured by centuries these irregularities would have little influence on the general average, but for short periods their influence may be great. A second source of discrepancy in the results lies in the inaccuracy of the surveys. Even where the sheet of water is so thin that the rock is visible thru it there is some liability to error, and where the topographer could see only the curved and changing surface of the rushing water his observations were necessarily somewhat indefinite. Two observers might in fact differ by several feet in their estimate of the actual position of the rock crest over which the water pours. The only results for shorter periods which it seems advantageous to place on record are those which use the map of 1875 in connection with the maps of 1842 and 1905. This approximately halves the whole period of sixty-three years, the earlier part being thirty-three years in length and the later part thirty years. By applying to these two divisions the methods already described for the whole period, and employing the same ordinates and the same limiting line, the following results were obtained:

*Rates of recession computed for various periods and by different methods.*

Limiting dates.	Length of period.	Average annual recession.	
		Computed by parallel ordinates.	Computed by areas.
	<i>Years.</i>	<i>Feet.</i>	<i>Feet.</i>
1842-1875.....	33	4.0	4.4
1875-1905.....	30	6.6	5.6
1842-1905.....	63	5.3	5.3

The indication is that during the thirty years following 1875 the lengthening of the gorge went on at a somewhat faster rate than during a similar period preceding that date. While it is quite possible that the apparent variation in the rate is sufficiently accounted

for by the irregularity of the breaking away of the limestone sill, it is also possible that the rate has been influenced by a special condition affecting the mode of recession. A change in the outline of the fall which was mentioned nearly a century ago as diminishing its resemblance to a horseshoe consisted in the development of an angle near the head of the curve and on the side toward Goat Island (Z, fig. 4). Within the last thirty years the recession has been especially rapid in that angle, and there has developed a deep recess or notch. This appears to have been occasioned by a local weakness of the limestone, presumably its subdivision by a belt of vertical joints. Within the notch the mode of recession has been so far modified that the upper layers of limestone have been removed before the lower, so that at certain stages of the process the water after falling from the crest has been caught by a shelf. The configuration can be better understood by an examination of Pl. I (p. 5), which is based on a photograph made in or near the year 1886. Whatever the method of erosion in the notch, it appears to be superadded to the general erosion by undermining, and an acceleration of the rate may plausibly be ascribed to it.

If we regard the general method of recession by the process of sapping or undermining as normal, and the influence of joint systems as exceptional and temporary, the rate of recession computed for the period from 1842 to 1875 should be accepted as normal and the best available for use in geologic computations; but this involves the assumption that the limestone ledge was not affected in other parts of the gorge by belts of weakness similar to the one which has been exposed during the last few decades. It seems to me better, on the whole, to assume that the limestone eroded between 1842 and 1905 is fairly representative, so far as strength is concerned, of all that portion of the limestone ledge in which the cataract has done its work.

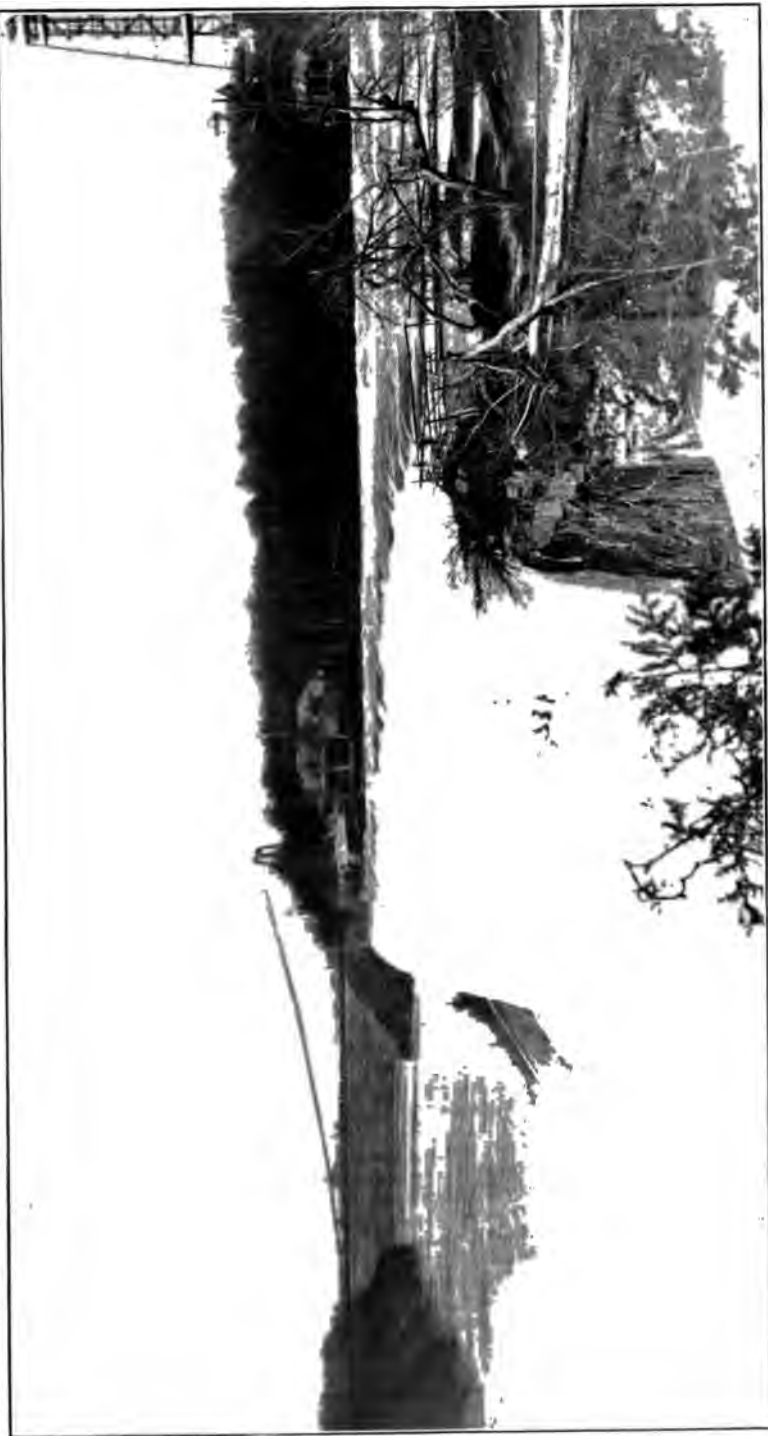
The maps of 1842 and 1905 represent the earliest and latest surveys, but do not include quite all the data worthy of consideration in this connection. A sketch by Basil Hall, made with the aid of a camera lucida, in 1827, has a claim for accuracy by no means to be disregarded. In the use of the camera lucida the draftsman sees the landscape as tho faintly pictured on a sheet of paper, and at the same time sees the pencil with which he traces its outlines. Before photography this method was the most accurate known for recording the outlines of a landscape, and in skilful hands it gives results of notable precision. There is much internal evidence that Captain Hall's sketches at Niagara were made with care and fidelity, and in view of these facts I have thought it worth while to endeavor to combine his record with the records by mapping. He tells us that his principal sketch of the Horseshoe Fall (Pl. III) was made from the upper veranda of Forsyth's Inn, on the Canadian shore, and the relation of the veranda to the inn is shown by a contemporary drawing by Mrs.





AMERICAN FALL IN 1827.

Copy of sketch by Capt. Basil Hall, made with camera lucida, from Goat Island. For explanation of broken line, *cf.* see page 19.



AMERICAN FALLS IN 1895.

Photograph from balcony just above view point of Pl. V.





Trollope.<sup>a</sup> The inn itself long since disappeared, but its position is still marked by the ruins of its foundations. Thru the courtesy of Mr. James Wilson, superintendent of Victoria Park, who caused the necessary scaffolding to be constructed, I was enabled, in 1895, to place a photographic camera within a few feet of the position once occupied by the camera lucida, and this position has also been located on the map of 1905 (Pl. II, p. 10). A comparison of the two pictures made from that position yielded the identification of a common point on one of the Three Sister Islands (S, Pl. III), and with the aid of the orientation thus secured it became possible to draw upon the modern map the line XY in fig. 4, representing a direction from Captain Hall's point of view tangent to the head of the Horseshoe Fall. It will be observed that this line passes very near to the head of the curve as drawn in the map of 1842, the implication being that at the head of the gorge very little recession had occurred in the fifteen years intervening between 1827 and 1842. I am not sure that this single line, obtained by so circuitous a method, should be allowed to influence the result based on two topographic surveys, but to whatever extent it is given consideration its tendency is to reduce the estimate of the annual rate.

#### THE AMERICAN FALL.

The recession of the American Fall is much slower than that of the Horseshoe. The sheet of water on its brink is comparatively thin, and the force the water acquires in falling is not sufficient to remove the larger of the limestone blocks broken from the ledge above. The blocks are therefore heaped at the base of the cliff and serve as a natural riprap to protect the shale against wear. (See Pl. XI, p. 22, and fig. 8, p. 23.) Since the Horseshoe Fall parted from the American, leaving it stranded at the side of the gorge, there has evidently been some falling away of the crest of the American Fall, else there would be no limestone blocks at its base. But as the talus increases in height it becomes more and more protective, and the rate of recession should theoretically diminish.

It has already been observed that the geologist's interest in the rate of recession applies primarily to the Horseshoe Fall, because the work of that fall makes the gorge longer. If the conditions of erosion had been uniform during the whole period of the excavation of the gorge the work of the American Fall would have little bearing on its time estimates, but the volume of the river has not always been so great as at present, and there were two epochs in the history of the gorge when the volume was very small. During those epochs the discharge of the whole river was probably not much greater than the present discharge thru the American channel, so that the conditions

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<sup>a</sup> Trollope, Mrs., *Domestic manners of the Americans*, vol. 2, London, 1832, frontispiece.

affecting erosion were somewhat similar to those illustrated by the American Fall. For this reason it is worth while to inquire at what rate the American Fall has receded since the first precise observations on its position and contour.

Traditional information as to changes in the American Fall is summarized by Lyell:<sup>a</sup>

The sudden descent of huge rocky fragments of the undermined limestone at the Horseshoe Fall, in 1828, and another at the American Fall, in 1818, are said to have shaken the adjacent country like an earthquake. According to the statement of our guide in 1841, Samuel Hooker, an indentation of about forty feet has been produced in the middle of the ledge of limestone at the lesser fall since the year 1815, so that it has begun to assume the shape of a crescent, while within the same period the Horseshoe Fall has been altered so as less to deserve its name.

The graphic record begins with two camera lucida sketches by Basil Hall, made in 1827. One was from Goat Island, near the southern end of the crest line, the other from a point on the American shore near the northern end of the crest line. His view points were so near to the fall that he was able to represent details too small to appear in the sketch of the Horseshoe Fall. The American Fall was also mapped with the same care as the Horseshoe in 1842, 1875, 1886, 1890, and 1905. Since the time of the daguerreotype the fall has been photographed from positions similar to those occupied by Basil Hall. and in 1895 I recovered his viewpoints as nearly as practicable for the sake of making photographs which might be compared with his camera-lucida sketches. To this end I visited the localities with his sketches in hand, and endeavored to determine the view points by comparing various details of the sketches with the landscape before me. His sketches and the photographs are compared in Pls. V-VI and VII-VIII.

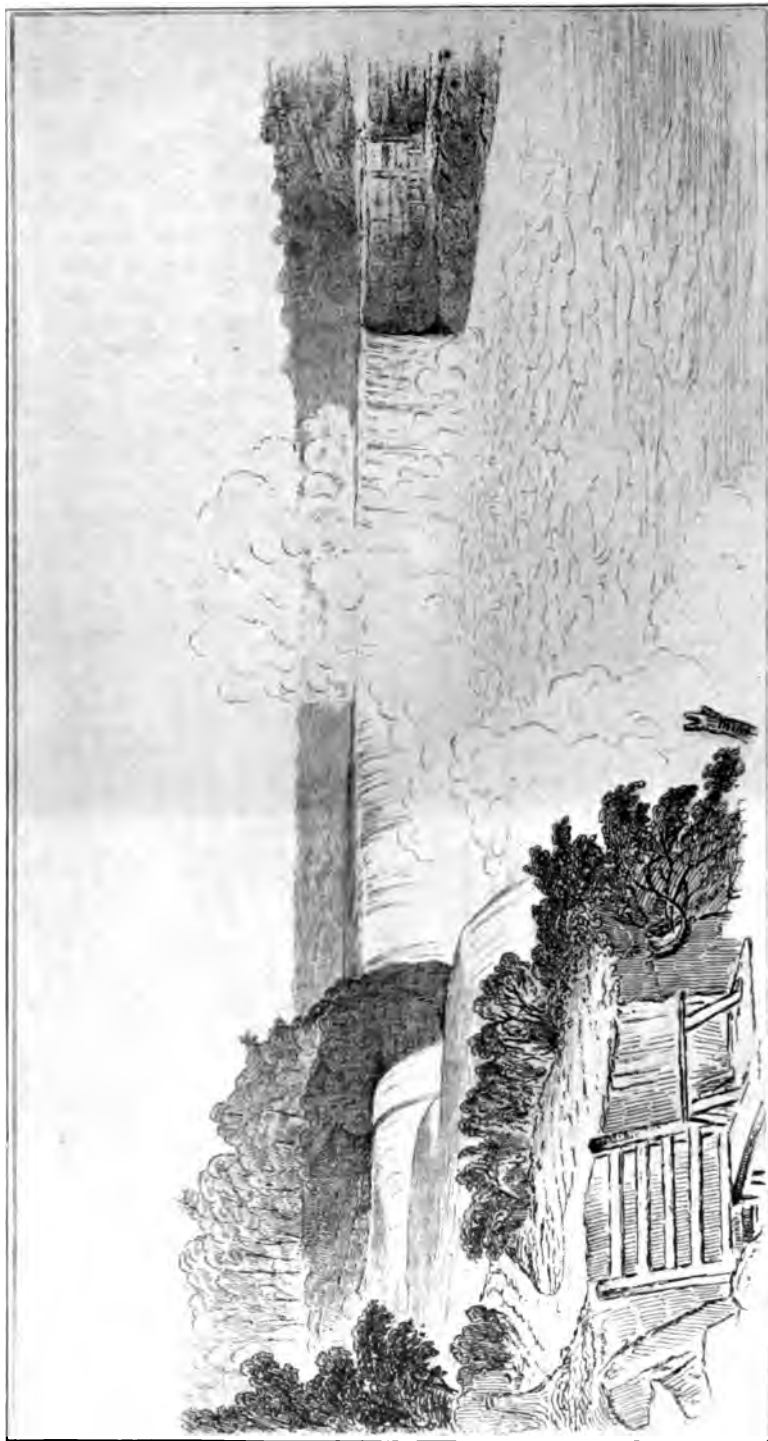
Examination of the combined map in Pl. II (p. 10) shows that the outlines recorded in 1875, 1886, 1890, and 1905 run closely together, the plotted lines intersecting one another at various points, while the line of 1842 coincides for only a part of the distance. A broad projection near the northern shore is indicated by the map of 1842 only, and that map also gives a more advanced position for the middle part of the crest line.

There is good reason to question the accuracy of the map of 1842, especially in the vicinity of the northern shore. The area there indicated outside the line of 1875 and later maps is 110 feet broad. As its position is close to Prospect Point, which has been a popular view point thru the entire period under consideration, the falling away of such a body of rock, either gradually or all at once, could not have escaped notice, but (so far as my reading goes) current literature, including the literature of the guidebooks, is silent in regard to it. In

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<sup>a</sup> Lyell, Charles, *Travels in North America*, vol. 1. London, 1845, p. 33.





AMERICAN FALLS IN 1827.

Engraved from a drawing by Capt. R. H. Wilkes, made on the day from August 10, 1827. The Horseshoe Falls is in the distance.



AMERICAN FALLS IN 1895.

Photograph from Prospect Point; a few feet to the left of the view point of Pl. VII. Note that the American Falls has changed little and the Horseshoe Falls much.



addition to this negative evidence, there is positive information in the Basil Hall sketches. Comparing his sketch from Goat Island (Pl. V) with my photograph made from approximately the same point in 1895 (Pl. VI), it will be seen that there is essential correspondence in the distant headlands along the river. By means of these headlands I was enabled not only to establish a definite relation between the two views, but also to correlate the sketch of 1827 with the map of the gorge made in 1875, and by the aid of that map with the various charts of the crest line. Thru these comparisons it is shown that if the crest line in 1827 had had the form indicated by the map of 1842, its profile would have the position indicated by the dotted line *A* in Pl. V, and the cataract would conceal the eastern half of the gorge vista. If the great salient did not exist in 1827, it could not have existed in 1842. The conclusion appears unavoidable that the map of 1842 is wholly erroneous in its delineation of that part of the crest line near Prospect Point.

As the Basil Hall sketches have thus served to discredit a portion of the map of 1842, it is in order to inquire whether they afford a substitute for the evidence ruled out. Once more using the vista down the gorge as the basis of correlation, and applying measurement to points recognized as identical, I have ascertained that the sketch of 1827 and the photograph of 1895 give to the extreme salient of the American Fall almost identically the same position. At that particular point the recession appears to be zero. Nearer than the salient, and appearing about one-fourth inch to the right of it, is a peculiar configuration of the crest line which seems to be common to the two views. In the photograph a dark wedge projects obliquely downward and toward the left, interrupting the body of white. In the sketch its position is occupied by a sweeping curve, less angular than the other lines representing the turn of the water. Making proper allowance for the fact that the water was unusually low in the summer of 1895, I think it quite possible that these features of the two pictures represent the same local and peculiar configuration of the rock of the crest, and the suggestion they give is that there has been no change whatever in the crest line of that portion of the American Fall since 1827.

The earliest good daguerreotype of the American Fall to which I have been able to assign a date is reproduced in Pl. IX. The gentleman who loaned me the daguerreotype appears in the picture as a child, and was able by that circumstance to fortify his memory and say that the view was taken in 1854 or 1855. Close comparison of the daguerreotype with the photograph reproduced in Pl. VIII, shows a large number of identical details ranged along the crest from the deepest reentrant to Luna Island, and proves that there was practically no recession in that part of the American Fall in the forty years from 1855 to 1895.



In Basil Hall's view from the American shore (Pl. VII) a number of points are sufficiently definite to be used in correlating the sketch with the map. Forsyth's Hotel appears on the bluff at the extreme right. The western edge of the Horseshoe Fall holds the same position as in 1842. The eastern edge of the Horseshoe Fall, or the right-hand profile of Goat Island, serves as another identification point, altho it has doubtless fallen away a few feet. The crest of the American Fall where it adjoins Goat Island and its interruption by Luna Island are somewhat indefinite objects by reason of the curvature of the water profile, but are nevertheless serviceable, especially as their stability is assured by the general agreement of records. The nearer profile of the American Fall is assumed on the evidence just cited to have the position assigned it by the maps of 1890 and 1905. These points all appear on the map (Pl. II, p. 10). The approximate position of the artist's viewpoint is suggested by the foreground, taken in connection with various allusions in the literature.

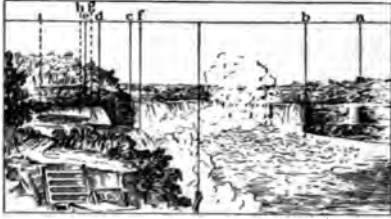


FIG. 5.—Basil Hall's sketch of American Fall from Prospect Point, with lines used in transferring its directions to map. Compare figs. 6 and 7 and Pls. VII and VIII.

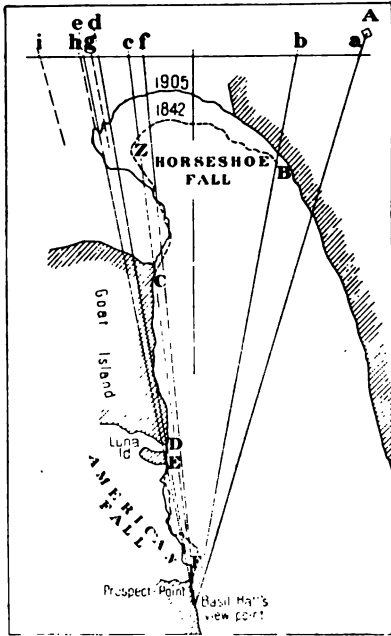


FIG. 6.—Map of Niagara Falls, with lines drawn to various points from the point occupied by Basil Hall in making a camera lucida sketch. Compare figs. 5 and 7 and Pl. VII. A, Forsyth's hotel; B, western edge of Horseshoe Fall; C, profile of Goat Island cliff; D, crest of American Fall at Goat Island; E, crest of American Fall at Luna Island; F, extreme salient on crest of American Fall.

horizontal direction of the object from the viewpoint. In order to show clearly the relations of the directions of the various objects, I drew from them a series of vertical lines by which their posi-

tion in fig. 5, to the right or left of a central vertical line depends on the

As the geometric method of making comparison between a picture and a map may not be familiar to all readers of this paper, I venture to explain the procedure in this case, adding that similar methods were employed in other comparisons to which allusion has already been made. It is evident that the distance of any object in the view, fig. 5, to the right or left of a central vertical line depends on the



THE AMERICAN FALL IN 1854 OR 1855.

From a daguerreotype. To be compared with the views on Pls. VII and VIII, but the view point is farther to the left and nearer the water. Many details of the crest of the American Fall are the same as in 1895, but the details of the Horseshoe Fall and the cliff profile of Goat Island differ from those of 1827 and 1895.



tions were projected against a horizontal line near the top of the sketch. Lines were also drawn on the map, fig. 6, from the assumed viewpoint to the corresponding objects, and an additional line was drawn in the general direction corresponding to the middle of the picture. Then at right angles to the last-mentioned line, and at a suitable distance ascertained by trial, a line was drawn intersecting all the direction lines. The map gives the projection of the various points on a horizontal plane; the sketch gives their projection on a vertical plane. The line last drawn represents the intersection of these two planes of projection. If the map and sketch are both accurate, then the points *a, b, c*, etc., on the map should be separated by the same spaces as the points *a, b, c*, etc., at top of the sketch. As a result of the trial a very close agreement was found—as close an agreement as could be expected in view of the indefiniteness of some of the points. This agreement serves to verify the determination of the viewpoint, and also to support the conclusion that the criticism previously made of the map of 1842 is valid.

Having thus established the relation of the sketch to the map, it was possible to transfer the directions of other points of the sketch to the map. Two reentrants and one salient of the fall were projected upward on the sketch, giving the points *g, h*, and *i*. These were transferred to the line on the map representing the intersection of projections, and lines were drawn from them to the viewpoint. These last-mentioned lines indicate on the map the directions of the corresponding features as recorded by the sketch of 1827, but do not show their distances from the viewpoint. They do not fix on the map the positions of the salient and reentrants, but assign certain limits to be observed in any attempt to chart the crest line as it was in 1827. They are represented on a larger scale in fig. 7. In a general way they indicate that there has been a small amount of recession since 1827 in various parts of the crest line. Such an inference, however, should not be

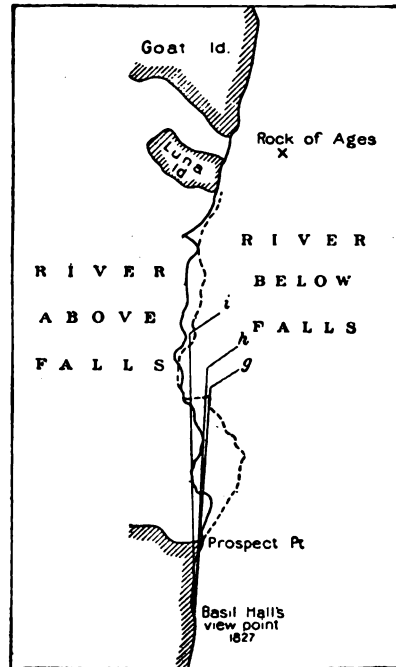


FIG. 7.—Plan of American Fall. A full line shows the crest as mapped in 1905; the broken line, as mapped in 1842; *i*, tangent to deepest reentrant as sketched by Basil Hall in 1827; *h*, tangent to reentrant nearer Prospect Point 1827; *g*, tangent to salient between two reentrants, 1827. Compare figs. 5 and 6 and Pl. VII.

made without qualification, because the indicated amount of recession is of the same order of magnitude as the errors of survey and other imperfections of the data.

To give the matter quantitative statement I have tried the experiment of assuming as correct the map of 1905 and the limitations inferred from the sketches of 1827, and then interpreting other data in such way as to afford the greatest plausible recession. A computation based on these assumptions gives an average total recession since 1827 of 19.7 feet and an average annual recession of 0.25 foot. This I regard as a maximum estimate. It is highly probable that the actual average rate of recession is less than this, and it may be much less. The idea that it is much less finds support in the identical appearance of one part of the crest in 1855 and 1905 and in the apparent identity of another part in 1827 and 1895.

The matter can be approached in another way. The distance thru which the Horseshoe Fall has retreated since it parted from the American Fall is about 2,500 feet. Allowing 5 feet per annum as the rate of recession, the parting took place about five hundred years ago. The condition of the American Fall at the time of separation may be inferred in a general way from an examination of the eastern part of the Horseshoe Fall at the present time (Pl. X). From Goat Island to a point about 500 feet westward the water is shallow, corresponding in average depth to that of the American Fall. Beyond that point it is comparatively deep. In the region of deep water the recession of the cataract is rapid, and the portion with shallow water is being left behind. At the base of that part of the fall where the water is shallow the descending stream does not plunge into the pool, but strikes a talus of rock fragments. These fragments are in part visible, and their existence is elsewhere inferred from the forms given to the spray by the reaction. It seems to me legitimate to infer that the American Fall at the time of its abandonment by the Horseshoe was not so advanced in position as to plunge into standing water, but had already retreated far enough to have acquired a talus above the level of the pool. At the present time the profile of the American Fall where its volume of water is greatest is approximately as shown in fig. 8. The edge of the main river is at S, 220 feet horizontally from the crest of the fall at C, the intervening space being occupied by a gently sloping talus of large limestone blocks, among which the water descends in a labyrinth of cascading torrents (Pl. XI). At the initial stage, when the American Fall was first separated, the position of its crest was probably at some point (I) between its present position and the outer edge of the visible talus. As sketched, I is 160 feet from C, and if the total retreat of the American Fall in five hundred years was 160 feet the average rate of recession was 0.32 foot per annum. Allowance should be made for difference in rate dependent



EASTERN PART OF HORSESHOE FALL ABOUT 1885.

Shows talus of limestone blocks. At the left, near Goat Island, there was a large rock fall in 1852.





AMERICAN FALL ABOUT 1885.

Shows taus of limestone blocks. Compare with fig. 8 in text, which represents the middle part of the fall.





on the gradual encroachment of the protective talus upon the exposed cliff of shale, so that during the earlier part of the period the retreat was more rapid than during the later part. The indication, therefore, is that the present rate of recession is considerably less than 0.32 foot per annum, a result in harmony with that based on the maps and sketches.

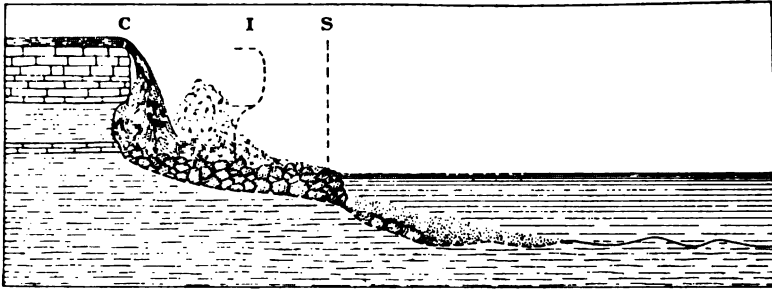


FIG. 8.—Profile and section of American Fall near its middle part. Compare fig. 1, also Pl. XI.

The assumptions underlying each of the estimates are factors of such importance that neither result can claim a high measure of precision. It appears to be safe to say that the present average rate of recession of the American Fall can not be so great as 0.5 foot per annum, and is probably as small as 0.2 foot per annum, or about one twenty-fifth of the rate of recession of the Horseshoe Fall.

#### THE MAP OF 1842.

The detection of an important error in the outline of the American Fall as mapped in 1842 tends naturally to bring in question all other results of the survey of that year. Inasmuch as the outline of the Horseshoe Fall as determined in 1842 is one of the most important data used in the computation of the rate of recession, it has been subjected to critical examination and all practicable checks have been applied.

The framework of the survey includes two stations or "trigonometrical points" on the American shore, three on Goat Island, and three on the Canadian shore. Those on Goat Island were connected, each with the next, by traverse lines, distances being measured by the surveyor's chain and courses observed by the surveyor's compass; so also were the two on the American shore. All other connections were made by compass bearings. From the 7 stations thus established the positions of 29 points on the crest lines were determined by intersections of compass bearings. In all the later surveys the bearings were presumably made with the engineer's transit or the plane-table alidade, instruments susceptible of much higher precision than the surveyor's compass; but in view of the shortness of the

distances the relative weakness of the surveyor's compass does not seem to me an important factor.

The stations and other points are indicated on the published map, and there is a "table of observations." With the aid of these data positions of points on the crest lines were replotted as a check on the accuracy of the compilation and engraving of the map. This work revealed three errors in the bearings as published, probably to be ascribed to copying or printing and not affecting the map. It indicated also that the points of the crest lines determined by intersection are not all accurately placed on the map, the errors amounting usually to a few feet, but not affecting the computed rate of recession.

Each of the crest-line points was originally located, as a rule, by the intersection of two bearings, but there are four points to which three bearings were taken. In the replotting of these points the check afforded by the third bearing was found to give a satisfactory result. The points which have the advantage of this check are all on the east side of the Horseshoe curve, and include the point at the angle of the Horseshoe in the position where the notch subsequently developed (Z in fig. 4, p. 14). For the remainder, or western part, of the Horseshoe curve there is no similar check, and the three located points of the crest line are so far apart as to give little mutual support. So far as the published data are concerned, these have no higher intrinsic authority than the two points on the American Fall which have been discredited by independent evidence.

The record of the Horseshoe Fall which stands nearest in time to the map of 1842 is Basil Hall's sketch from the Forsyth Hotel (Pl. III, p. 15), the interval being fifteen years. The general form of the crest line is the same in map and sketch, and the tangent based on the sketch is so related to the mapped crest line (fig. 4, p. 14) as to indicate some recession between the dates of the sketch and the map, but the amount of recession is less than would be expected.

The factors bearing on the estimate of the rate of recession are not so related that rigid mathematical methods can be applied to their discussion. The conflict of data and the mutual support of data can be weighed only by nonmathematical methods, and the result of their study is an opinion rather than a decision. The general tenor of the evidence, including the five surveys and the Basil Hall sketch, leaves no question that the annual rate of recession has been about 4 or 5 feet. If full authority be ascribed to the map of 1842, the estimated annual rate of recession is 5.3 feet. If full authority be ascribed to the tangent line based on the sketch of 1827, the estimated rate is about 1 foot less. It is my opinion that the map affords the better record. Giving to it the greater weight and to the tangent a smaller weight, I think the best practicable estimate of the rate is between 5.3 and 4.2 feet, but nearer to the former; and I select 5 feet partly

because a statement in even feet avoids the implication of high precision which might be suggested by a decimal. As an estimate of the average rate of recession during the period of definite observation, I think this can not be in error more than 1 foot.

#### SUMMARY AND CONCLUSION.

The data for computing the rate of recession of Niagara Falls include surveys of the crest line made in 1842, 1875, 1886, 1890, and 1905, and camera-lucida sketches made in 1827. During the period covered by these data the local conditions affecting the rate of recession have not differed to an important extent from the natural conditions. The present and prospective diversions of water for economic uses interfere with the course of nature and may be expected to modify the rate of recession. The natural rate of recession of the Horseshoe Fall is desired by geologists in connection with estimates of the age of the river. The geologic bearing of a rate modified by human agency is less direct. The rate of recession of the American Fall is of interest to geologists because somewhat representative of the river's activity in gorge making when the volume of water was much less.

The rate of recession of the Horseshoe Fall, or the rate of lengthening of the Niagara gorge, during the sixty-three years from 1842 to 1905 is found to be 5 feet per annum, with an uncertainty of 1 foot. For the thirty-three years from 1842 to 1875 the rate was apparently slower than for the thirty years from 1875 to 1905. The rate of recession of the American Fall during the seventy-eight years from 1827 to 1905 was less than 3 inches per annum.

The time consumed in the recession of the falls from the escarpment at Lewiston to their present position, or the age of the river, is not here estimated. It can not properly be computed without taking account of all conditions, local and temporary, affecting the rate of recession, and some of those conditions have varied greatly from point to point and from time to time.

## REPORT OF SURVEY OF CREST LINE OF NIAGARA FALLS.

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By W. CARVEL HALL.

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In obedience to instructions from Mr. H. M. Wilson, geographer of the United States Geological Survey, issued in consequence of a plan of cooperation with Mr. Henry A. Van Alstyne, State engineer of New York, a survey of the crest line of Niagara Falls was made by me in June, 1905. The determination of the present crest line of the falls was desired in order that, by comparison with maps of earlier dates, the changes could be determined and the rate of recession computed.

Surveys of the falls of which we have record have been made as follows: In 1842, under the direction of Prof. James Hall, State geologist of New York;<sup>a</sup> in 1875, by the United States Lake Survey, published in the form of a chart on the scale of 1:2,500;<sup>b</sup> in 1886, by Prof. R. S. Woodward, then chief geographer of the United States Geological Survey;<sup>c</sup> and in 1890, by Mr. A. S. Kibbe, assistant engineer, under the direction of Mr. John Bogart, State engineer of New York.<sup>d</sup> A survey was also made in 1904 by the Electrical Development Company and Prof. J. W. Spencer, but the map is not yet published.

The great majority of the monuments recovered or established by Mr. Kibbe in his survey of 1890 were found to be in good state of preservation, but a few, one unfortunately an important one, have disappeared. Appended hereto are tables giving descriptions of the various monuments recovered or used, together with their coordinates and the distances between them. In addition to these, there were used in the work temporary stations at Prospect Point, Hennepin View, Stedman Bluff, Rock of Ages, center of south chord of the steel arch bridge, and the cupola of Table Rock House.

In the survey of 1842 the relations of a few points were fixed by traversing with compass and chain, and the remainder of the work was done by compass, the positions of points on the crest being determined by intersection. In the surveys of 1875, 1886, and 1890

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<sup>a</sup> See Nat. Hist. of New York, pt. 4, Geology, 1843, pp. 402-404.

<sup>b</sup> Listed by the Lake Survey as Chart No. 48.

<sup>c</sup> Results published in Seventh Ann. Rept. Comrs. of State Reservation at Niagara, Albany, 1891.

<sup>d</sup> *Idem*.

directions were observed by transit instead of compass. In the 1904 work an ingenious method was used, suggested, it is thought, by Mr. Goodwin, of the Electrical Development Company. He carefully measured the elevation of the crest of the falls and also of certain triangulation points on the Canadian bluff. Then, observing at the same instant for direction and for the amount of the "dip-angle," he computed the distance from point of observation to the crest at various places.

Any of these methods seems quite suitable for the determination of the crest at well-marked points, but the upper curve of the Horseshoe Falls has no well-marked points for observation, besides being masked by spray, and the methods adopted require considerable computation and use mainly descriptive matter for identifying even the most important features. As stated by Professor Woodward in his report already referred to—

The points on the crests of the Falls determined by the 1886 survey varied considerably in respect to clearness of definition and ease and precision of fixture. Some of them were well defined exposures of bare rock; some were less well-defined portions of rock seen through the curved sheet of falling water; some were indentations in such sheets, well defined from any point of view but presenting different aspects from different points of view; and some were ephemeral sprays whose identification from different points of view was a matter of difficulty. \* \* \* the probable error on the crest line does not on the average exceed  $\pm 1.5$  feet. A much greater probable error must be assigned, however, to the upper part of the Horseshoe Falls.

Mr. Kibbe in his 1890 report states that "for favorable locations on the crest of the Falls the probable error is  $\pm 1.0$  foot, while along the deep-water portion of the Horseshoe Falls it may be somewhat greater."

In view of these facts, a different method was adopted in making the present survey, namely: T. P. No. 1 (at Prospect Point) and "Terrapin" (at Terrapin rocks) were accepted from Mr. Kibbe's survey as initial points, and were plotted to a scale of 1 inch = 200 feet on a plane-table sheet. From these as a base all existing triangulation points were relocated, and with some newly selected points formed the basis of the survey. Along the American Falls and at the ends of the Horseshoe Falls, by means of photographs and careful descriptions, numerous features were recognized, intersected, and plotted, and the details of the map were continuously compared with the corresponding natural features and their positions checked. Thus any discrepancy between earlier surveys and the present work were at once seen and examined and were thoroly tested and proved before the work was accepted, the majority of locations being determined from six to eight stations. For the upper portion of the Horseshoe Falls a 30-inch searchlight of very high candlepower, partially masked, was used to mark on the crest of the falls successive small brilliant spots of light, which were simultaneously

located by four transits and which it is believed should result in an accurate demarcation of that part of the crest.

An anomalous condition of affairs appears to be disclosed by the survey, similar to that noted in earlier work, viz, that certain points on the crest line have advanced instead of receded. This has heretofore been explained as due to discrepancies in the field work or inaccuracies in the delineation of the crest line between determined points. Doubtless these explanations hold good in all the surveys, past as well as present, but there is one cause of advancement and ultimate recession which does not appear to have been commented on, and which, after close questioning of numerous old residents and careful examination of the situation, I think is a very active element. It might here be emphasized that by the plane-table method used any discrepancy occurring was at once noticed and the new position most carefully checked.

In addition to the erosion caused by the spray and the chemical action of the water on the underlying shales, there is a well-marked change in the crest line, due to the forcing off of large blocks of rock from the crest itself.

Approximately 20 feet below the upper level there is a water-bearing seam in the limestone, particularly well marked at Goat Island and above the Ontario Power Company's new power house. I am informed that in the winter immense icicles form from this seam at various points where it is not noticeable in summer, owing to the quick evaporation. At about this same elevation there are at present on the American Falls four secondary cascades, or, as termed by the late Thomas V. Welch, superintendent of the State reservation, "bustles." In my judgment these can only have been caused by the slipping off of a mass of rock from the crest, sliding to some extent on the water-bearing seam and gradually prest out by the force of the water and ice in slowly widening cracks upstream till, overbalancing, they fall, leaving the "bustles." The present survey, I believe, has caught some of these masses as they are being forced out.

This theory was discussed with a number of the best-informed inhabitants, and while they agreed that large blocks did occasionally fall from the crest, instancing several examples, they claimed that in each case the fall was a sudden one, unaccompanied by any previous pushing forward of the mass. This forward motion, however, must be a very slow one, and would not be noticed, while the final fall of the rock would be assumed to be the beginning and end of the action.

The accompanying plan of the survey (Pl. II, p. 10) shows the existing crest line by a solid line broken only where the flow of water is interrupted by overhanging rocks, and shows the earlier surveys by

means of broken lines. Only those triangulation points which are permanently marked are indicated. The shore lines and the various permanent buildings are a compilation of all the records.

In the last few years the brink of the falls has been curtailed on the Canadian side by a wall 495 feet long, completely shutting off the water for that distance, and thereby reducing the length of the Horseshoe Falls by about one-sixth. In recent years five power companies have spent large sums of money to divert a portion of the water now flowing over the falls to their power houses, the total power at present chartered being about one-fifth of all available.

I desire to acknowledge my indebtedness for material assistance rendered in the work to Mr. Edward H. Perry, superintendent of the State reservation at Niagara; to Mr. James Wilson, superintendent of the Queen Victoria Niagara Falls Park; to Mr. B. F. R. Paine, general manager of the Ontario Power Company, and to Mr. J. W. Kellogg, manager of the marine sales department of the General Electric Company.

## TABLES.

The first of the following tables includes artificial monuments and other permanent reference points connected with the triangulations of the surveys. The designation, description, and location of each are given, together with its coordinates as referred to Topographic Point No. 1 of the survey of 1842. The coordinates, with the exception of Semaphore, are taken from Mr. Kibbe's table in the Seventh Annual Report of the Commissioners of the State Reservation at Niagara, pages 105 to 107.



*List of permanent reference points.*

Designation.	Description.	Coordinates.	
		<i>Feet.</i> 0.0	<i>Feet.</i> 0.0
T. P. No. 1.....	1842, 1890, 1905. Stone monument 5 inches square on Prospect Point 1 inch below sod. 138 feet from southwest corner stone house at head of incline, 145 feet from northwest corner of same, and 15 feet from iron lamp-post.		
M.....	1890, 1905. Stone monument 5 inches square, 1 foot below surface, with 6-inch terra-cotta tile over it, set on bluff in extreme edge of gravel walk at Porters Bluff, just east of path to the Terrapin Rocks.	S. 1,929.1	W. 1,206.5
T. P. No. 6.....	1842, 1890, 1905. Stone monument 5 inches square, in path following top of bluff along southern shore of Goat Island. This monument projects about 7 inches and is marked "6" on side.	S. 2,370.7	W. 990.4
Terrapin.....	1886, 1890, 1905. Brass bolt 1 inch in diameter set in prominent square rock about 4 feet high, word "Terrapin" cut in top of rock at end of wooden walk to platform where Terrapin Tower formerly stood. Believed to coincide very nearly with point used in 1875.	S. 2,044.2	W. 1,465.5
Loretto.....	1886, 1890, 1905. Cross surmounting cupola of Loretto Convent, also brass screw one-half inch in diameter in the deck of cupola directly under center of cross.	S. 3,671.4	W. 3,623.4
N.....	1890, 1905. Brass bolt 1 inch in diameter near superintendent's office, Canadian side, set in the rock 1 foot under surface of lawn and surrounded by 4-inch terra-cotta tile, 34.9 feet from southwest corner main building and 18.3 feet from southeast corner same.	N. 1,021.9	W. 1,462.1
B.....	1890, 1905. Brass bolt 1 inch in diameter set in rock between pipe railing and brink, in Canadian reservation, 30.2 feet from northeast corner of railing at "Ramblers Rest."	N. 788.5	W. 1,451.8
C.....	1890, 1905. Stone monument 6 inches square between pipe railing and brink, in Canadian reservation, 25.4 feet northerly from pipe of drinking fountain south of "Ramblers Rest."	N. 193.9	W. 1,751.6
D.....	1890, 1905. Stone monument 6 inches square, between pipe railing and brink, in Canadian reservation, a little south of "Inspiration Point."	S. 458.2	W. 2,090.4
I.....	1890, 1905. Stone monument 6 inches square, marked "I" on side, set in prominent projecting point at top of bluff on Canadian side about 317 feet southeasterly from south gable of Ontario power house, now in course of construction (June, 1905).	S. 1,602.5	W. 2,773.2
K.....	1890. Stone monument like last, about 400 feet southwardly along bluff.	S. 1,934.7	W. 2,996.2
L.....	1890. Stone monument like last, about 490 feet southwardly along bluff.	S. 2,338.0	W. 3,261.3
Semaphore.....	Center of 8-inch iron pipe sunk several feet in the ground on west bank railway cut about opposite Clarke Springs.	S. 2,496.6	W. 3,507.2
O.....	1890. Stone monument 6 inches square set into slope at Stedmans Bluff; covered in 1900 by dry stone wall; references as on line between T. P. No. 1 and a nail in root of large leaning oak tree 21.41 feet southwest and 49.7 feet from nail in root of basswood tree.	S. 989.1	W. 597.1





## CLASSIFICATION OF THE PUBLICATIONS OF THE UNITED STATES GEOLOGICAL SURVEY.

[Bulletin No. 306.]

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CHARLES D. WALCOTT, DIRECTOR

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MANUAL  
OF  
TOPOGRAPHIC METHODS

BY

HENRY GANNETT



WASHINGTON  
GOVERNMENT PRINTING OFFICE  
1906

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# MANUAL OF TOPOGRAPHIC METHODS.<sup>a</sup>

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By HENRY GANNETT.

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## CHAPTER I.

### INTRODUCTION.

#### OBJECT OF THIS MANUAL.

The object of this manual is a description of the topographic work, instruments, and methods of the United States Geological Survey, primarily for the information of the men engaged in this work. It is not intended to be an elementary treatise on surveying, as it presupposes a knowledge of the application of mathematics to surveying equivalent to that obtained in our professional schools. Neither is it intended to be a general treatise on topographic work, altho it may, to a certain extent, supply the need of such a work.

Surveys are made for two widely different purposes. One consists in laying down upon the ground certain geometric figures, such as a town plat or the alignment of a railroad or an aqueduct. The other purpose is the making of maps, or miniature representations of the country. While the instruments and operations are to some extent the same in both, the purpose and the results are very different.

Most books on surveying have in view mainly, if not entirely, the first of these two purposes, operations incident to the making of maps being more or less slighted. Herein the author will attempt to describe the most approved methods of surveying as applied to the production of topographic maps, whether on large or small scales and whether of a high or a low degree of accuracy, including rough reconnaissance as well as accurate and detailed surveys.

#### CLASSES OF MAPS.

A map is a representation, in plan, of a part of the earth's surface. The variety of maps is legion, depending upon the class of features or phenomena which they represent or which is made prominent on them. Thus there are geologic maps, zoologic maps, botanic maps,

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<sup>a</sup> This manual was first published in 1893 as Monograph XXII of the United States Geological Survey.

cadastral maps (which represent, primarily, property lines), political maps, etc. For all these it is essential that there be a base upon which the specific phenomena can be represented. This base should show certain topographic features, as streams and other bodies of water, railroads, towns, and cities; while some sort of convention to represent the relief of the ground is usually a valuable adjunct.

From the point of view of scale, those maps which show only topographic features may be grouped in two classes: First, those on small scales, say 10 or more miles to an inch, which may be properly called geographic maps and which are usually compiled from other maps on larger scales; and, second, those on large scales, which may properly be called topographic maps. Maps of the latter class are often made directly from surveys, and being the first product of these surveys, are known as mother maps, because they constitute the source from which other maps are compiled. In this work are described the methods of surveying and the preparation of maps on large scales—maps which can be conveniently made directly from surveys.

#### TOPOGRAPHIC MAPS.

A topographic map should represent rivers, creeks, lakes, ponds, and all other bodies of water, together with coast lines; all artificial features that are of sufficient prominence to be represented on a scale, such as roads, boundary lines, cities, towns, villages, houses, and, in case of large-scale maps, fences and other such objects, bridges, fords, dams, canals, aqueducts, etc., and the relief of the ground—its hills, valleys, mountains, canyons, etc.

The relief of features can be shown by different methods, which may be classed as qualitative and quantitative. Qualitative methods show relief by shading, either by means of crayon or color or by means of hachure lines—lines which are drawn in the direction of the slope and which by their relative heaviness and closeness produce the effect of shading. These methods merely show the degree of slope; they give no information regarding the absolute amount of relief, or elevation above sea level. They have been in extensive use thruout the world, but their use is now rapidly diminishing.

#### CONTOURS.

The method of expressing relief now commonly in use, which has largely replaced the methods described above, is that known as the contour method. Contours are lines drawn at equal elevations above sea level, every point upon a contour being, or being supposed to be, at the elevation indicated by that line. These contours would become shore lines if the level of the sea were raised successively to the heights indicated by them. This method is con-

fessedly not so graphic as the others, but it has this great advantage, that it expresses absolute elevations; the height above the datum (usually sea level) of any portion of the area represented can be read directly from the contours. Moreover, it expresses the slopes, since where they are steep the contours must necessarily be close together, and where they are gentle the contours must be far apart. Modern maps, as a rule, are contour maps.

The contour interval is the vertical scale of the map. This should be proportioned to the horizontal scale and to the degree of relief of the country. With the same horizontal scale the contour interval would naturally be large for a country of bold relief and small for one of slight relief, since in the former case the same difference of altitude is of much less importance than in the latter case.

### SCALES.

The scale of a map is the proportion which exists between the dimensions of the map and those of the area which it represents. It is designated in several ways:

By the number of miles or of feet represented by an inch on the map.

By what is called a fractional scale, the numerator representing a linear unit of the map and the denominator expressing the number of such units in a corresponding distance on the ground, as  $\frac{1}{100000}$ .

By a bar scale, wherein a measure on a map is marked with its corresponding measure on the ground.

On many maps all of these forms of designating the scale are given.

The scale of a map should be chosen so as to represent as much detail and as great accuracy as are needed for the purpose for which the map is made. It should be neither larger nor smaller than is required to suit this purpose. For a small amount of detail a large scale is unnecessary, while too small a scale does not allow the legible representation of the detail required. Thus the statement of the scale of a map should afford an idea of its accuracy and detail.

### CULTURAL FEATURES.

The list of cultural features, or works of man, which should be represented on a map depends upon the scale of the map and the purposes for which it is made. A general topographic map, which is designed for use for many years without revision, should contain comparatively few of these features—only those which are likely to be permanent and which are of importance to the community rather than to individuals. These features change with considerable rapidity, and keeping the map revised involves the expenditure of much labor and money if many such features are represented. For these reasons their introduction should be restricted so far as is compatible

with the usefulness of the map. For maps on large scales and for temporary purposes the list may be enlarged to any extent and in any desired direction, but the matter of inclusion and exclusion of these features should be considered carefully in view of all attendant circumstances. It is very easy so to clutter a map with useless information as to obscure its really valuable features.

#### PLAN OF THE MAP OF THE UNITED STATES.

##### DIVERSITY OF CONDITIONS.

The field upon which the United States Geological Survey is at work is diversified. It comprizes broad plains, some of which are densely covered with forests, while on others trees are absent; high and rugged mountains, plateaus, and low, rolling hills. In some regions its topographic forms are on a grand scale; in other regions the entire surface is made up of minute detail. Some parts of the country are densely populated, as much so as any other region on the earth, while in other parts of the country great areas are almost without settlement. Geologically, portions of the country are extremely complex, requiring, for the elucidation of geologic problems, maps in great detail, while other areas are very simple.

It is obvious that, because of this diversity of conditions, maps of different areas should vary in scale, and that with such diversity in natural conditions and in scale there must be differences in the methods of work employed. The system which is found to work advantageously in the high mountain regions of the West is more or less inapplicable to the low, forested plains of the Mississippi Valley and the Atlantic Plain.

##### SCALE.

The scales which have been adopted for the publication of the map are 1:62500, or very nearly 1 mile to an inch; 1:125000, or very nearly 2 miles to an inch; and 1:250000, or very nearly 4 miles to an inch.

The following considerations have determined the selection of the above scales: They are believed to be sufficiently large to admit of the representation of all the details necessary to a picture of the country which shall show the proper relations of its features and shall be of the greatest possible service for industrial and scientific uses consistent with other requirements to be mentioned hereafter. These scales are also sufficiently large to enable geologists to present the details of nearly all geologic phenomena. The map represents the country in sufficient detail to admit of the selection of general routes for railroads and other public works, and of the indication of boundary lines in such way that their positions may be recognized upon the ground. On the other hand, the scales are not so large as to prevent the rep-

resentation of a considerable area on a single sheet, so that the relation between different regions can be seen at a glance.

A map on a larger scale would be more expensive and would require longer time for its completion. On the scales adopted the map of the United States, excluding Alaska, will cost about twenty-five million dollars and at the present rate of progress will require fifty years for its completion.

There is another objection to increasing the scale beyond that absolutely necessary, viz: Such a map, to be of value, must undergo revision at frequent intervals in order that changes in culture and possibly in natural features due to natural or artificial agencies may be incorporated. The larger the scale the more frequently such revision would be necessary, and hence the labor and expense of keeping such a map up to date would be greatly increased.

#### CONTOUR INTERVAL.

The relief of the earth's surface is now represented on maps almost entirely by contour lines, or lines of equal elevation. Until a comparatively recent date this relief, secondary in importance only to the horizontal location, or the plan, has not been expressed quantitatively.

The contour intervals which have been adopted for the map of the United States are as follows: For the scale of 1:62500 the intervals range from 5 to 50 feet; for the scale of 1:125000 the range is from 10 to 100 feet; and for the scale of 1:250000 the interval is 100, 200, or 250 feet.

#### FEATURES REPRESENTED.

The experience of European nations tends in the direction of reducing the number of features which should be placed on a map. In the preparation of the map of the United States it has been decided to go even beyond the present practise of European countries in this regard, and to show only natural features of such magnitude that the scale warrants their representation and cultural features of general or public importance. Under these limitations the map will show cities, towns, and villages; roads, railroads, and other means of communication; bridges, ferries, tunnels, fords, canals, and acequias; and boundaries of civil divisions. Fences, property lines, and other objects of kindred nature will not be represented. The reasons for excluding private culture are apparent: First, because such features are not of sufficient general interest to pay the cost of surveying or representing them; second, because they change rapidly, and in order to keep the maps up to date constant resurveys and republication would be required; and, third, because their number and complexity confuse the map and render its more important features less intelligible.

## SIZE OF SHEETS.

The atlas sheets are designed to be of approximately the same size—17½ inches in length by 12 to 15 inches in width, depending upon the latitude. Those on the same scale cover equal areas expressed in units of latitude and longitude; that is, each sheet on the 4-mile scale covers 1 degree of latitude by 1 degree of longitude; each sheet on the 2-mile scale, 30 minutes of latitude and 30 minutes of longitude; and each sheet on the 1-mile scale, 15 minutes of latitude and 15 minutes of longitude. The sheets are thus small enough to be conveniently handled, and if bound they form an atlas of suitable size. From the fact that each sheet covers either a full degree or a certain fractional part of a degree, its position with relation to adjacent sheets and to the area of the country may be readily ascertained.

## CHAPTER II.

### METHODS AND CLASSIFICATIONS OF WORK.

#### GENERAL METHODS.

Every map is essentially a sketch controlled by locations. It follows that the work of making a map consists of two parts—that of fixing these locations, which is done by means of surveying instruments and is geometric, and that of sketching, which is done by the hand and eye and is artistic. This general description applies to the making of all maps, whatever their scales, their accuracy, their degree of detail, or the character of the organization making them. The methods and instruments employed in fixing the locations vary with different scales, kinds of country, and the ideas of the organizations engaged in the work.

Altho the sketching is all that appears on the finished map, a description of the methods of making the locations must of necessity form the bulk of a volume descriptive of surveying methods, for it is the essential part of the work. This volume will treat only of the most approved methods and instruments now used in topographic surveying; time and space will not be wasted upon methods or instruments that have been tried and discarded.

#### CORRECTNESS OF MAPS.

The correctness of a map depends upon four elements: Accuracy of location, number of locations per square inch of map. distribution of locations, and quality of the sketching.

#### ACCURACY OF LOCATION.

The greatest accuracy attainable is not always desirable, because it would not be economical. The highest economy is in properly subordinating means to ends, and it is not economical to do work of greater accuracy than is needed—for instance, to execute triangulation of geodetic refinement for the control of maps on small scales. The quality of the work should be such as to insure against errors of sufficient magnitude to be appreciable on paper. While errors in triangulation tend to balance one another, still they are liable to accumulate, and this liability must be guarded against by maintaining a somewhat higher degree of accuracy than would be required



for the location of any one point. It is not difficult to meet this first condition of accuracy of maps. The maximum allowable error of location may be set at 0.01 of an inch on the scale of publication. This admits of an error upon the ground, on a scale of 1:62500, not greater than 50 feet, and proportionally on other scales.

#### NUMBER OF LOCATIONS PER SQUARE INCH OF MAP.

The second condition of correctness for the proper control of the work is not easily defined. The requirements differ with the character of the country. A region of great detail and of abrupt features requires more control than one of greater uniformity, of gentle slopes, or of large features, so that no general rule can be laid down. Furthermore, it depends upon the quality of the sketching. With indifferent sketching a greater number of locations is required in order to bring the map to the requisite quality. Examples of the amount of control upon maps are scarce, since little has been published on this subject.

There are two general methods of locating stations and points for the correction of the sketch—one by angular measurements (triangulation) and the other by measurement of direction and distance, or what is known popularly as the traverse or meander method. In ordinary practise work may be done by either of these methods, or they may be used in conjunction. The former method can be carried on with the plane table, with various forms of the theodolite, with a compass, or, indeed, with any angle-reading instrument. The latter method can be pursued with the same instruments, supplemented by various forms of odometers, chain, steel tape, stadia, etc., for the measurement of distance. The first method, whenever it can be used economically, is the more accurate and, as a rule, the more rapid, and locations thus made are likely to be of greater service and to be distributed more uniformly than if made by the other method. It can be used economically where the country presents relief and where either natural or artificial points for location exist in sufficient numbers and are well distributed. These conditions are satisfied almost everywhere in the western mountain regions, where peaks, summits of hills, plateau points, buttes, etc., furnish an abundance of natural points for stations and locations. It can be used to a considerable extent, tho not with the same ease and economy, in the Appalachian Mountains; but in this region it is necessary to supplement it extensively by traverse lines, especially in tracing the courses of streams in valleys. It can be used in the hill country of New England, where objects of culture, such as churches, houses, etc., furnish plenty of signals. On the other hand, thruout the whole extent of the Atlantic and Gulf plains and the Mississippi Valley, where the country is level, or nearly so, and is covered with forests,

the traverse method of surveying must be resorted to. In these parts of the country there are no sharp natural objects, and extended views can not be obtained. For many reasons the second method of obtaining locations is inferior to the former. It is inferior not only in accuracy but in the facilities which it affords for sketching the country, and it should be so regarded and should be adopted only when it becomes necessary or when the former method can not be applied economically. For convenience traverse lines are generally run along roads or trails and thus the best points for commanding views of the country are avoided rather than sought, and there is danger lest the topographer neglect the areas lying between the roads. On account of the errors incident to running a traverse, it is necessary that in this class of work frequent locations be made by triangulation for checking and thereby eliminating the errors.

As a rule, triangulation locations are selected points chosen because each controls positions in a certain area. On the other hand, traverse locations, as a rule, are not chosen for their control value, but only for intervisibility on roads. Furthermore, the great majority of traverse stations are of no service whatever beyond carrying the line forward, so that in estimating the total amount of control in a certain area where the control is made up wholly or in part of traverse lines less weight should be given to them than to locations by triangulation.

#### DISTRIBUTION OF LOCATIONS.

The third element of accuracy is a point concerning which it is equally difficult to speak definitely. Other things being equal, the distribution should be uniform over the area, but it will necessarily vary with the character of the surface. In general, in mountain regions, locations by angular measurements are frequent and follow the ranges or ridges, but in valleys such locations are few in number, being supplemented by traverses.

The subject of locations may be treated from another point of view—that of the features to be represented. All points for locations should be chosen for the purpose of guiding the sketch of certain features. Streams, roads, and other linear features ordinarily can be best and most economically located by means of traverse lines; that is, by establishing by traverse a series of points along their courses. Houses can usually be best located by intersection, either in connection with the secondary triangulation or from a traverse.

The location of contour lines requires more than a passing mention. At the outset a very common impression should be corrected. It is generally supposed by those unacquainted with the subject that contours are located by finding and fixing points directly on each contour and sketching the contour between these points. This

method, however, is very rarely employed, and then only in the most detailed surveys. In the topographic surveys of all nations, except the English Ordnance Survey, a different method is employed, or, rather, a different selection of points is made. For location and height measurement salient points are selected, such as the summit of a hill, the shoulder of a spur, the foot of a slope, the bed of a valley. The contours are then sketched with reference to these points. By judicious selection of such points comparatively few locations and height measurements serve to locate the intervening contours with sufficient accuracy. On the whole, the results are quite as good as if points had been located immediately on the contours, and the work of location is vastly reduced.

#### QUALITY OF SKETCHING.

The fourth of the elements of the correctness of the map depends on the artistic sense of the topographer, on his ability to see things in their proper relations, and on his facility in transferring his impressions to paper.

Every map, whatever its scale, is a reduction from nature and is generalized, and consequently departs from the original in greater or less degree. The smaller the scale the greater the generalization, and the further the map must depart from nature the less can it resemble the ground which it represents. Bends in streams, petty gulches, canyons, and hills must be omitted. Contours must cross such omitted gulches instead of turning up and down their courses. A searching examination of the map would therefore show countless places where the contours are widely out of place, owing simply to this generalization. To specify that the contours of a map must nowhere be in error more than a contour interval, for instance, is to require an impossibility.

The education of the topographer therefore consists of two parts—the mathematical and the artistic. The first may be acquired largely from books, but this book knowledge must be supplemented by practise in the field. The second, if not inherent, can be acquired only by long experience in the field, and by many can never be acquired in high degree. In fact, the sketching makes the map, and it should be done by the best topographer in the party, usually the chief, whenever practicable.

#### CLASSIFICATION OF WORK.

The work involved in making a map usually comprizes several operations, which in practise may be more or less distinct from one another. They are as follows:

- (1) The location of the map upon the earth's surface, by means of astronomic observations.

(2) The horizontal location of points. This is usually of three grades of accuracy: Primary triangulation, or primary traverse in cases where triangulation is not feasible; secondary triangulation for the location of numerous points within the primary triangulation; and ordinary traverse, for the location of details.

(3) The measurement of heights, which usually accompanies the horizontal location, and which may, similarly, be divided into three classes, in accordance with the degree of accuracy.

(4) The sketching of the map.

Nearly all of the geometric work of surveying—the work of location—is executed with few instruments, as follows:

Theodolites, of a powerful and compact form, used in the primary control.

Wye levels.

Plane tables, with telescopic alidades of the best type, used for secondary triangulation and height measurements.

Plane tables of simple form, with ruler alidades, compasses, or solar attachment, used for traversing and minor triangulation.

Aneroids, for the measurement of details of heights.

These instruments, with which nine-tenths of the work is done, will be described in their proper places with such fulness of detail as seems necessary.

Other instruments, such as transits, surveyor's theodolites, compasses, hand and Abney levels, telemeters, chains, and tapes, are occasionally used. Most of these instruments, which are figured and described in nearly all works on surveying, are assumed to be well known to the readers of this manual, and will therefore receive no special attention.

## CHAPTER III.

### ASTRONOMIC DETERMINATION OF POSITION.

#### OBJECT AND IMPORTANCE OF ASTRONOMIC DETERMINATION

The object of astronomic determinations of position is to locate the map upon the earth's surface. They are made also for the purpose of checking and correcting positions determined by primary triangulation and primary traverse.

With regard to the checking of primary triangulation by astronomic determinations, it should be understood that in the case of a single determination the work by triangulation is far more accurate than that by the astronomic method, even when made under the best circumstances. It is, therefore, desirable to introduce checks of this kind upon primary triangulation only when the latter has been carried for a long distance—200 or 300 miles, for instance—in the course of which there may have accumulated errors greater than those incident to astronomic work.

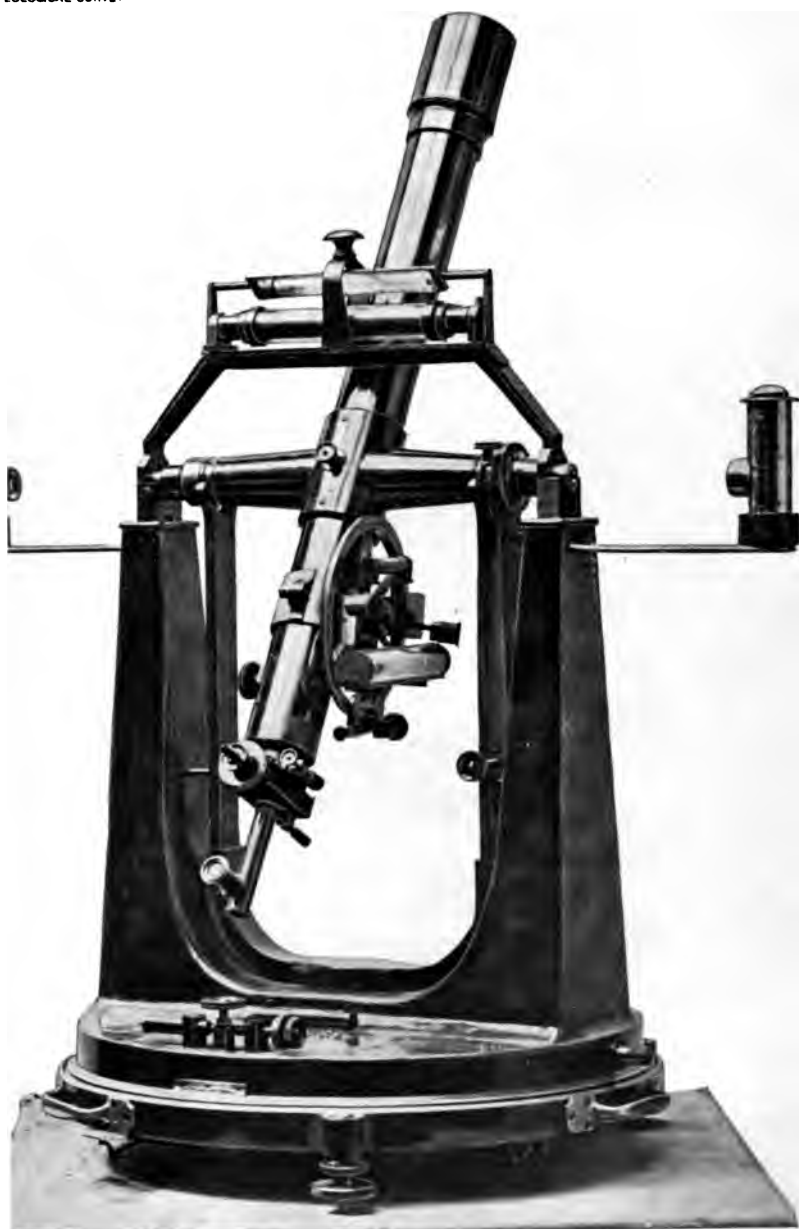
The case is different with primary traverse. The great number of courses required in this work affords an opportunity for the accumulation of error much greater than can arise in triangulation, and consequently it is desirable to introduce more frequent checks. It may be said that, in general, such work should be checked at every 100 miles.

As has been suggested, the best astronomic determinations are none too good for the control of maps. Indeed, certain errors incident to this work, some of which as yet can not be corrected, may be of magnitude sufficient to show upon the map. It is necessary, therefore, in these determinations to use the best instruments and the most refined methods known to modern science, in order to reduce all avoidable errors to a minimum.

Determinations of position that have been made by the United States Coast and Geodetic Survey, the United States Lake Survey, or the Mississippi River Commission, whether by astronomic work or by triangulation, may be utilized for the above purposes.

#### DEFINITIONS.

Sidereal time is the time indicated by the stars, a sidereal day being the time which elapses between two passages of the vernal equinox across the meridian. Solar or apparent time is the time



ASTRONOMIC TRANSIT AND ZENITH TELESCOPE.



measured by the sun's apparent movement, or the revolution of the earth with reference to the sun; and since the earth revolves at a differing rate in different portions of its orbit, the solar days are not of equal length. A mean day is the average solar day; mean time differs from solar time by an amount which varies with the time of year, and which, under the name "equation of time," is given in the Nautical Almanac. Mean time differs from sidereal time by about a day in the course of a year, or about four minutes in each day, the mean day being longer than the sidereal day. To convert a given date of mean time into sidereal time it is necessary to obtain, from the Nautical Almanac, the sidereal time at noon immediately preceding the date in question. Then the interval after noon, expressed in mean time, is converted into sidereal time, and the result added to the sidereal time of mean noon. Local time, whether sidereal, solar, or mean, is the time of the locality as distinguished from the time of any other locality. It must be distinguished from standard time, which is the local time of only certain meridians.

The right ascension of the sun or a star is the sidereal time which has elapsed between the passage of the vernal equinox and the star across the meridian. It is commonly expressed in hours, minutes, and seconds.

Declination is the angular distance of a heavenly body north or south of the equator. It is plus when north and minus when south of the equator.

The zenith distance of a heavenly body equals its declination minus the latitude of the place of observation.

Latitude is determined by what is known as Talcott's method, by measuring the differences of zenith distance at culmination of two stars which culminate on opposite sides of the zenith.

Longitude is determined by telegraphic comparison of local time at two stations, the longitude of one of which is known. This involves the determination of the errors of the clocks or chronometers used, which is done by observation of transits of stars across the meridians of the places of observation.

#### ASTRONOMIC TRANSIT AND ZENITH TELESCOPE.

A single instrument is used for the determination of both latitude and time. This is a combination of the transit and the zenith telescope. The instruments in use by the United States Geological Survey were made by Saegmüller and embody the latest improvements. (See Pl. I.) The circular base rests upon three leveling screws. Upon this circular base the whole instrument can be made to revolve when using it as a zenith telescope. A circle is graduated around the base, having a micrometer screw for slow motion, for making settings and adjusting the instrument in azimuth. The frame of the



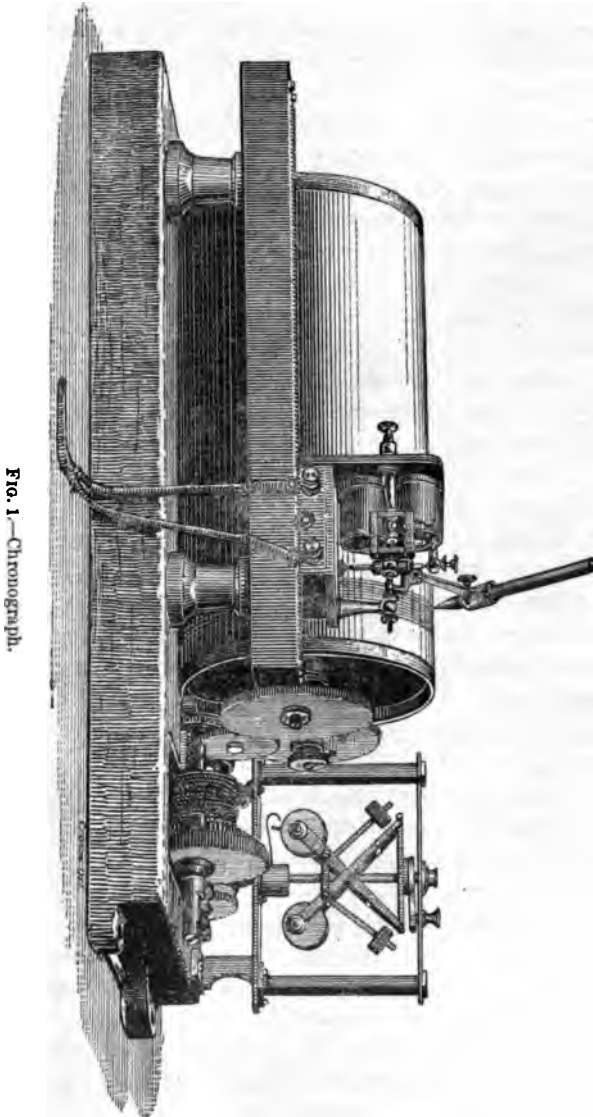
instrument is cast in one piece, and the standards are hollow in order to reduce the weight of the upper part. The telescope has a focal distance of 27 inches and a clear aperture of 2.5 inches. Its magnifying power with diagonal eyepiece is 74 diameters. The length of the axis of the telescope is 16 inches. For use as a zenith telescope the instrument is equipped with a vertical circle reading by vernier to 20 seconds, attached to which is a delicate level. In the focus of the object glass there is, besides the ordinary reticule for use in transit work, a movable thread, which is moved by means of a micrometer screw, by which measurements of differences of zenith distances are made. It is furnished with direct and diagonal eyepieces, the latter of which is commonly used in astronomic work.

For use as a transit instrument the telescope is equipped with a delicate striding level for measuring the inclination of the pivots and a reversing apparatus for turning the telescope in the wyes. The reticule, as the stationary threads in the focus of the instrument are called, consists of five threads for observing the transits of stars. The reticule is illuminated by means of bull's-eye lamps, the light from which comes thru the hollow axis of the telescope and is reflected by a mirror placed at the intersection of the telescope with its axis.

#### CHRONOGRAPH.

The chronograph is used for the purpose of recording the time of transits of stars as observed with the transit instrument. It may be popularly characterized as an instrument for measuring time by the yard. It consists essentially of a drum upon which is wound a strip of paper and which is kept in revolution by a train of clockwork controlled by an escapement. A pen carried on a small car, which is moved very slowly in a direction parallel to the axis of the cylinder, traces a spiral line upon the paper on the drum. This pen is held in place by a magnet carried on the car, and as long as the current from the battery passes thru the coil and thus holds the armature, the pen traces an unbroken spiral line. If the current is suddenly broken and restored, the armature is set free for an instant and a jog is made in the line traced. The battery commonly used in connection with this outfit is the ordinary zinc copper, and copper sulfate battery, of which four cells are usually required. The ordinary dry battery can also be used and is much more convenient. With this apparatus break-circuit chronometers are used. These differ from ordinary chronometers in that they are arranged to break an electric circuit automatically at regular intervals. Those in use by the United States Geological Survey break the circuit every two seconds, and the end of the minute is indicated by breaking at the fifty-ninth as well as the fifty-eighth and sixtieth seconds. When one of these chronometers is connected with a battery and a chronograph is put in the same

uit, the beginning of every even second is recorded upon the chronograph by a jog on the paper, and the distance between the jogs each case therefore represents two seconds. The observer at the trument is provided with a telegraph key, which may also be put the circuit with the chronometer and chronograph, and as a star



ur the meridian crosses a thread in the telescope he records that t by pressing on the key, which makes a record upon the chronoph along with the record of the chronometer. An illustration of the form of chronograph in use by the United ates Geological Survey is shown in fig. 1.

## FIELD WORK.

Since the observations for latitude and longitude, tho different, are made with the same instrument, at the same time, and by the same party, certain parts of the work apply equally to both determinations and may be described once for all.

In the selection of a station care must be taken to avoid a locality where for any cause the ground is liable to be seriously jarred, as, for instance, proximity to a railroad track or to a street over which heavy wagons pass. The station should have a clear view from the southern horizon thru the zenith to the northern horizon. It is desirable to locate at a convenient distance from a telegraph office, as it is necessary to bring in a wire from such office for the purpose of comparing chronometers. If possible, the station should be selected on a public reservation, in order that the permanence of the monument which is to be erected to mark the spot may be assured. In any event a locality in which such a monument is likely to be disturbed should be avoided.

The support of the instrument should consist of a brick, stone, or concrete pier sunk fully 3 feet in the ground and rising to the requisite height. Upon this should be placed a block of stone well set in mortar for the immediate support of the instrument. The chronograph may be set up on an ordinary table. Over all should be erected a wall tent with a slit in the roof, closed by flaps, which can be opened when observing. The instrument should be set up on the pier, collimated, and leveled, the verticality of the threads should be tested as accurately as possible, and then it should be pointed upon the pole star. This places it somewhere near the meridian. Then taking the time of transit of a star which culminates close to the zenith, and comparing this time with the right ascension of the star, a sufficiently close approximation to the clock error is obtained for use in placing the instrument in the meridian. The instrument should then be turned in azimuth to point upon some close circumpolar star approaching upper or lower culmination, the instrument being moved in azimuth with the tangent screw, so as to keep the star under the middle wire to the instant of culmination. If this is done accurately at the first attempt the instrument is placed nearly in the meridian and is ready for work, but it commonly happens that more than one trial is required before the meridian is reached. In any case the result should be verified by a second star before proceeding with the observations.

## OBSERVATIONS FOR LATITUDE.

As preliminary to this work it is necessary to prepare a list of pairs of stars, the two stars of each pair having such zenith distances that they will culminate at nearly equal distances from the zenith, one to the north and the other to the south of it. Such a list can be prepared from the Safford Catalog of the Wheeler Survey. For this it is necessary to know the approximate latitude of the station and the right ascensions and the declinations of the stars. The zenith distance of a star is equal to its declination minus the latitude of the place of observation. The stars of each pair must be observed consecutively, and therefore those stars should be selected which culminate as nearly as possible together, leaving only a sufficient interval of time between them for setting the instrument.

On the approach of the first star of the pair to the meridian the instrument should be set for it, using the vertical circle for that purpose, and setting the spirit level upon the vertical circle as nearly level as possible. Then, as the star traverses the field of the telescope, the movable thread in the reticule should be kept upon it by means of the micrometer screw until it crosses the middle vertical thread. Then the micrometer and the two ends of the level bubble should be read and recorded. Without disturbing in the slightest degree the setting of the telescope, the entire instrument should be turned 180 degrees upon its bedplate, when it will point north of the zenith at the same angle at which it formerly pointed south, or vice versa, as the case may be, and will be set for the other star on the opposite side of the zenith. As this approaches culmination it should be followed with the micrometer as before until it reaches the middle thread, then the readings of the micrometer and of the level should be recorded as before, whether changed or not.

This constitutes the observations upon a single pair of stars. For the determination of latitude twenty such pairs of stars should be observed each evening, if possible, and the same pairs of stars should also be observed on other evenings, assuming it to be possible. The following example, taken from observations at Rapid, S. Dak., shows a portion of the star list and the form of record:

## MANUAL OF TOPOGRAPHIC METHODS.

*List of stars for observation with zenith telescope.*[Station: Rapid, S. Dak. Approximate latitude:  $44^{\circ} 05'$ .]

Name or number. Safford's Catalog.	Mag.	Class.	R. A.	Dec.	Zen. dist.	Setting.
7 Lacertæ....	4.0	A A	<i>h. m.</i> 22 27	<i>° '</i> 49 43	<i>° '</i> 5 38 N.	<i>° '</i> 5 37 N.
10 Lacertæ....	5.0	A A	22 34	38 29	5 36 S.	
1539.....	6.5	B	22 41	45 37	1 32 N.	1 27 N.
1551.....	6.5	A	22 47	42 42	1 23 S.	
1565.....	6.5	C	22 52	38 42	5 23 S.	5 22 S.
1579.....	5.0	A	22 59	49 26	5 21 N.	
1600.....	6.0	A	23 08	56 34	12 29 N.	12 19 N.
1633.....	6.7	B	23 18	31 56	12 09 S.	
1676.....	5.6	A	23 42	67 12	23 07 N.	23 05 N.
1686.....	6.5	A	23 47	21 03	23 02 S.	
1702.....	4.5	A	23 52	24 32	19 33 S.	19 31 S.
1722.....	6.5	B	24 00	63 35	19 30 N.	

*Example of record.*

[Station: Rapid, S. Dak. Date: November 9, 1890. Instrument: Fauth combined transit and zenith telescope No. 534. Observer: S. S. G. Recorder: A. F. D.]

Star name or number.	N. or S.	Micrometer reading.	Diff.	Level.		(N+S) -(N'+S').	Remarks.
				N.	S.		
7 Lacertæ....	N.	Rev. 26.256	- 2.204	Div. 39.9	Div. 16.7	Div. +56.6	
10 Lacertæ....	S.	24.052		26.5	49.7	-76.2 -19.6	
1539.....	N.	30.432	-10.337	42.0	18.7	+60.7	
1551.....	S.	20.095		21.9	45.0	-66.9 - 6.2	
1565.....	S.	25.164	+ 1.539	14.1	37.6	-51.7	Faint.
1579.....	N.	26.703		38.1	15.0	+53.1 + 1.4	Distinct.
1600.....	N.	32.214	-16.181	37.5	14.1	+51.6	
1633.....	S.	16.033		19.9	43.1	-63.0 -11.4	Faint.
1676.....	N.	26.656	- 8.972	51.0	28.0	+79.0	
1686.....	S.	17.684		17.0	39.6	-56.6 -22.4	
1702.....	S.	25.345	+ 1.623	18.0	40.9	-58.9	
1722.....	N.	23.722		36.0	13.2	+49.2 - 9.7	

## REDUCTION OF LATITUDE OBSERVATIONS.

Before proceeding with the reduction of latitude observations it is necessary to investigate the constants of the instrument and to ascertain the value of a division of the head of the micrometer screw and of a division of the latitude level.

## MEASUREMENT OF A DIVISION OF THE HEAD OF THE MICROMETER SCREW.

The value of a division of the head of the micrometer screw is measured by observing the transits of some close circumpolar star, when near elongation, across the movable thread, setting the thread repeatedly at regular intervals in advance of the star, and taking the time of its passage with the reading of the micrometer. The precaution should be taken to read the latitude level occasionally and correct for it if necessary. This correction, which is to be applied to the observed time, is equal to one division of the level, in seconds of time, divided by the cosine of the declination of the star and multiplied by the level error, the average level reading being taken as the standard.

Determination of value of one revolution of micrometer belonging to Z. T. No. 534, by observations on 51 Cephei near eastern elongation, November 15, 1890, Rapid, S. Dak.

$\log \cot \delta \dots 87^\circ 12' 50'' \text{ } s = 8.6871826$   
 $\log \tan \phi \dots 44 \quad 04 \quad 45 = 9.9860340$   
 $\log \cos \iota_0 \dots 87 \quad 17 \quad 57 = 8.6732206$   
 $\log 1 \text{ div. } (=1.33) \dots = 0.12385$   
 $a. c. \log \cos \delta \dots = 1.31324$   
 $a. c. \log 15 \dots = 8.82391$   
 $\log 1.82 \dots = -0.26110$

$h \quad m \quad s$   
 $\iota_0 = 5 \quad 49 \quad 11.3$   
 $\alpha = 6 \quad 49 \quad 20.4$   
 $T_0 = 1 \quad 00 \quad 08.6$   
 $\delta T = \quad \quad -8.0$

chron. time long. = 1 00 00.6

Time from elongation.		Correc- tion for change of level		Reduction to mean level.		Correc- tion for T.		Reduced time.		Revolutions.		Time for nine revolutions.	
m.	s.	m.	s.	m.	s.	m.	s.	m.	s.	m.	s.	m.	s.
17	04.0	+1.5	+1.3	18	48.3	+0.9	+1.4	00	40	31.0 and 30.5	22.0 21.5	15	37.7 36.6
16	16.0	+0.5	+0.3	15	24.7	+0.8	+0.7	42	55.4	23.5 23.0	20.5 19.5	34.4 33.2	34.4 33.2
15	24.7	+0.3	+0.0	14	28.7	+0.5	+0.5	43	43.9	23.5 23.0	19.5 17.5	40.2 38.9	40.2 38.9
14	28.7	+0.0	-0.3	13	33.2	+0.6	+0.6	44	36.9	23.5 23.0	17.5 16.5	36.8 35.5	36.8 35.5
13	33.2	-0.5	-0.8	12	38.0	+0.8	+0.8	45	32.4	23.5 23.0	16.5 15.5	37.5 36.2	37.5 36.2
12	38.0	-1.1	-1.1	11	43.5	+0.8	+0.8	46	07.5	23.5 23.0	15.5 14.5	38.2 36.9	38.2 36.9
11	43.5	-1.1	-1.1	10	49.3	+0.6	+0.6	47	56.2	23.5 23.0	14.5 13.5	39.1 37.8	39.1 37.8
10	49.3	-1.1	-1.1	9	55.8	+0.6	+0.6	48	49.3	23.5 23.0	13.5 12.5	40.2 38.9	40.2 38.9
9	55.8	-1.0	-1.0	8	62.4	+0.6	+0.6	49	40.1	23.5 23.0	12.5 11.5	41.3 40.0	41.3 40.0
8	62.4	-0.9	-0.9	7	69.1	+0.6	+0.6	50	30.1	23.5 23.0	11.5 10.5	42.4 41.1	42.4 41.1
7	69.1	-0.9	-0.9	6	76.0	+0.6	+0.6	51	20.1	23.5 23.0	10.5 9.5	43.5 42.2	43.5 42.2
6	76.0	-0.9	-0.9	5	83.0	+0.6	+0.6	52	10.1	23.5 23.0	9.5 8.5	44.6 43.3	44.6 43.3
5	83.0	-0.9	-0.9	4	90.0	+0.6	+0.6	53	00.1	23.5 23.0	8.5 7.5	45.7 44.4	45.7 44.4
4	90.0	-0.9	-0.9	3	97.0	+0.6	+0.6	54	50.1	23.5 23.0	7.5 6.5	46.8 45.5	46.8 45.5
3	97.0	-0.9	-0.9	2	104.0	+0.6	+0.6	55	40.1	23.5 23.0	6.5 5.5	47.9 46.6	47.9 46.6
2	104.0	-0.9	-0.9	1	111.0	+0.6	+0.6	56	30.1	23.5 23.0	5.5 4.5	49.0 47.7	49.0 47.7
1	111.0	-0.9	-0.9	0	118.0	+0.6	+0.6	57	20.1	23.5 23.0	4.5 3.5	50.1 48.8	50.1 48.8
0	118.0	-0.9	-0.9	0	125.0	+0.6	+0.6	58	10.1	23.5 23.0	3.5 2.5	51.2 50.0	51.2 50.0
0	125.0	-0.9	-0.9	0	132.0	+0.6	+0.6	59	00.1	23.5 23.0	2.5 1.5	52.3 51.1	52.3 51.1
0	132.0	-0.9	-0.9	0	139.0	+0.6	+0.6	60	50.1	23.5 23.0	1.5 0.5	53.4 52.2	53.4 52.2
0	139.0	-0.9	-0.9	0	146.0	+0.6	+0.6	61	40.1	23.5 23.0	0.5 0.5	54.5 53.3	54.5 53.3
0	146.0	-0.9	-0.9	0	153.0	+0.6	+0.6	62	30.1	23.5 23.0	0.5 0.5	55.6 54.4	55.6 54.4
0	153.0	-0.9	-0.9	0	160.0	+0.6	+0.6	63	20.1	23.5 23.0	0.5 0.5	56.7 55.5	56.7 55.5
0	160.0	-0.9	-0.9	0	167.0	+0.6	+0.6	64	10.1	23.5 23.0	0.5 0.5	57.8 56.6	57.8 56.6
0	167.0	-0.9	-0.9	0	174.0	+0.6	+0.6	65	00.1	23.5 23.0	0.5 0.5	58.9 57.7	58.9 57.7
0	174.0	-0.9	-0.9	0	181.0	+0.6	+0.6	66	50.1	23.5 23.0	0.5 0.5	60.0 58.8	60.0 58.8
0	181.0	-0.9	-0.9	0	188.0	+0.6	+0.6	67	40.1	23.5 23.0	0.5 0.5	61.1 59.9	61.1 59.9
0	188.0	-0.9	-0.9	0	195.0	+0.6	+0.6	68	30.1	23.5 23.0	0.5 0.5	62.2 61.0	62.2 61.0
0	195.0	-0.9	-0.9	0	202.0	+0.6	+0.6	69	20.1	23.5 23.0	0.5 0.5	63.3 62.1	63.3 62.1
0	202.0	-0.9	-0.9	0	209.0	+0.6	+0.6	70	10.1	23.5 23.0	0.5 0.5	64.4 63.2	64.4 63.2
0	209.0	-0.9	-0.9	0	216.0	+0.6	+0.6	71	00.1	23.5 23.0	0.5 0.5	65.5 64.3	65.5 64.3
0	216.0	-0.9	-0.9	0	223.0	+0.6	+0.6	72	50.1	23.5 23.0	0.5 0.5	66.6 65.4	66.6 65.4
0	223.0	-0.9	-0.9	0	230.0	+0.6	+0.6	73	40.1	23.5 23.0	0.5 0.5	67.7 66.5	67.7 66.5
0	230.0	-0.9	-0.9	0	237.0	+0.6	+0.6	74	30.1	23.5 23.0	0.5 0.5	68.8 67.6	68.8 67.6
0	237.0	-0.9	-0.9	0	244.0	+0.6	+0.6	75	20.1	23.5 23.0	0.5 0.5	69.9 68.7	69.9 68.7
0	244.0	-0.9	-0.9	0	251.0	+0.6	+0.6	76	10.1	23.5 23.0	0.5 0.5	71.0 69.8	71.0 69.8
0	251.0	-0.9	-0.9	0	258.0	+0.6	+0.6	77	00.1	23.5 23.0	0.5 0.5	72.1 70.9	72.1 70.9
0	258.0	-0.9	-0.9	0	265.0	+0.6	+0.6	78	50.1	23.5 23.0	0.5 0.5	73.2 72.0	73.2 72.0
0	265.0	-0.9	-0.9	0	272.0	+0.6	+0.6	79	40.1	23.5 23.0	0.5 0.5	74.3 73.1	74.3 73.1
0	272.0	-0.9	-0.9	0	279.0	+0.6	+0.6	80	30.1	23.5 23.0	0.5 0.5	75.4 74.2	75.4 74.2
0	279.0	-0.9	-0.9	0	286.0	+0.6	+0.6	81	20.1	23.5 23.0	0.5 0.5	76.5 75.3	76.5 75.3
0	286.0	-0.9	-0.9	0	293.0	+0.6	+0.6	82	10.1	23.5 23.0	0.5 0.5	77.6 76.4	77.6 76.4
0	293.0	-0.9	-0.9	0	300.0	+0.6	+0.6	83	00.1	23.5 23.0	0.5 0.5	78.7 77.5	78.7 77.5
0	300.0	-0.9	-0.9	0	307.0	+0.6	+0.6	84	50.1	23.5 23.0	0.5 0.5	79.8 78.6	79.8 78.6
0	307.0	-0.9	-0.9	0	314.0	+0.6	+0.6	85	40.1	23.5 23.0	0.5 0.5	80.9 79.7	80.9 79.7
0	314.0	-0.9	-0.9	0	321.0	+0.6	+0.6	86	30.1	23.5 23.0	0.5 0.5	82.0 80.8	82.0 80.8
0	321.0	-0.9	-0.9	0	328.0	+0.6	+0.6	87	20.1	23.5 23.0	0.5 0.5	83.1 81.9	83.1 81.9
0	328.0	-0.9	-0.9	0	335.0	+0.6	+0.6	88	10.1	23.5 23.0	0.5 0.5	84.2 83.0	84.2 83.0
0	335.0	-0.9	-0.9	0	342.0	+0.6	+0.6	89	00.1	23.5 23.0	0.5 0.5	85.3 84.1	85.3 84.1
0	342.0	-0.9	-0.9	0	349.0	+0.6	+0.6	90	50.1	23.5 23.0	0.5 0.5	86.4 85.2	86.4 85.2
0	349.0	-0.9	-0.9	0	356.0	+0.6	+0.6	91	40.1	23.5 23.0	0.5 0.5	87.5 86.3	87.5 86.3
0	356.0	-0.9	-0.9	0	363.0	+0.6	+0.6	92	30.1	23.5 23.0	0.5 0.5	88.6 87.4	88.6 87.4
0	363.0	-0.9	-0.9	0	370.0	+0.6	+0.6	93	20.1	23.5 23.0	0.5 0.5	89.7 88.5	89.7 88.5
0	370.0	-0.9	-0.9	0	377.0	+0.6	+0.6	94	10.1	23.5 23.0	0.5 0.5	90.8 89.6	90.8 89.6
0	377.0	-0.9	-0.9	0	384.0	+0.6	+0.6	95	00.1	23.5 23.0	0.5 0.5	91.9 90.7	91.9 90.7
0	384.0	-0.9	-0.9	0	391.0	+0.6	+0.6	96	50.1	23.5 23.0	0.5 0.5	93.0 91.8	93.0 91.8
0	391.0	-0.9	-0.9	0	398.0	+0.6	+0.6	97	40.1	23.5 23.0	0.5 0.5	94.1 92.9	94.1 92.9
0	398.0	-0.9	-0.9	0	405.0	+0.6	+0.6	98	30.1	23.5 23.0	0.5 0.5	95.2 94.0	95.2 94.0
0	405.0	-0.9	-0.9	0	412.0	+0.6	+0.6	99	20.1	23.5 23.0	0.5 0.5	96.3 95.1	96.3 95.1
0	412.0	-0.9	-0.9	0	419.0	+0.6	+0.6	100	10.1	23.5 23.0	0.5 0.5	97.4 96.2	97.4 96.2
0	419.0	-0.9	-0.9	0	426.0	+0.6	+0.6	101	00.1	23.5 23.0	0.5 0.5	98.5 97.3	98.5 97.3
0	426.0	-0.9	-0.9	0	433.0	+0.6	+0.6	102	50.1	23.5 23.0	0.5 0.5	99.6 98.4	99.6 98.4
0	433.0	-0.9	-0.9	0	440.0	+0.6	+0.6	103	40.1	23.5 23.0	0.5 0.5	100.7 99.5	100.7 99.5
0	440.0	-0.9	-0.9	0	447.0	+0.6	+0.6	104	30.1	23.5 23.0	0.5 0.5	101.8 100.6	101.8 100.6
0	447.0	-0.9	-0.9	0	454.0	+0.6	+0.6	105	20.1	23.5 23.0	0.5 0.5	102.9 101.7	102.9 101.7
0	454.0	-0.9	-0.9	0	461.0	+0.6	+0.6	106	10.1	23.5 23.0	0.5 0.5	104.0 102.8	104.0 102.8
0	461.0	-0.9	-0.9	0	468.0	+0.6	+0.6	107	00.1	23.5 23.0	0.5 0.5	105.1 103.9	105.1 103.9
0	468.0	-0.9	-0.9	0	475.0	+0.6	+0.6	108	50.1	23.5 23.0	0.5 0.5	106.2 105.0	106.2 105.0
0	475.0	-0.9	-0.9	0	482.0	+0.6	+0.6	109	40.1	23.5 23.0	0.5 0.5	107.3 106.1	107.3 106.1
0	482.0	-0.9	-0.9	0	489.0	+0.6	+0.6	110	30.1	23.5 23.0	0.5 0.5	108.4 107.2	108.4 107.2
0	489.0	-0.9	-0.9	0	496.0	+0.6	+0.6	111	20.1	23.5 23.0	0.5 0.5	109.5 108.3	109.5 108.3
0	496.0	-0.9	-0.9	0	503.0	+0.6	+0.6	112	10.1	23.5 23.0	0.5 0.5	110.6 109.4	110.6 109.4
0	503.0	-0.9	-0.9	0	510.0	+0.6	+0.6	113	00.1	23.5 23.0	0.5 0.5	111.7 110.5	111.7 110.5
0	510.0	-0.9	-0.9	0	517.0	+0.6	+0.6	114	50.1	23.5 23.0	0.5 0.5	112.8 111.6	112.8 111.6
0	517.0	-0.9	-0.9	0	524.0	+0.6	+0.6	115	40.1	23.5 23.0	0.5 0.5	113.9 112.7	113.9 112.7
0	524.0	-0.9	-0.9	0	531.0	+0.6	+0.6	116	30.1	23.5 23.0	0.5 0.5	115.0 113.8	115.0 113.8
0	531.0	-0.9	-0.9	0	538.0	+0.6	+0.6	117	20.1	23.5 23.0	0.5 0.5	116.1 114.9	116.1 114.9
0	538.0	-0.9	-0.9	0	545.0	+0.6	+0.6	118	10.1	23.5 23.0	0.5 0.5	117.2 116.0	117.2 116.0
0	545.0	-0.9	-0.9	0	552.0	+0.6	+0.6	119	00.1	23.5 23.0	0.5 0.5	118.3 117.1	118.3 117.1
0	552.0	-0.9	-0.9	0	559.0	+0.6	+0.6	120	50.1	23.5 23.0	0.5 0.5	119.4 118.2	119.4 118.2
0	559.0	-0.9	-0.9	0	566.0	+0.6	+0.6	121	40.1	23.5 23.0	0.5 0.5	120.5 119.3	120.5 119.3

Determination of value of one revolution of micrometer belonging to Z. T. No. 534, by observations on 51 Cephei near eastern elongation, November 15, 1890, Rapid, S. Dak.—Continued.

Time of observation (recorded on chronograph sheets)		Micrometer revolutions		Level		Reduction to mean level		Correction for change of level		Time from elongation = 1.		Correction for i.		Reduced time			
h	m	s	Dir.	Dre.	N	S	Dir.	Dre.	N	S	s.	s.	s.	h.	m.	s.	
33	22	4	38	3	17	3	55	6									
34	18	8															
35	07	1															
36	01	2															
36	32	4	38	3	17	3	55	6									
37	41	2															
38	07																
39	25	9															
40	17	9															
41	10	0															
(mist)																	
42	52	8	38	0	17	0	55	0									
43	44	8															
44	42																
45	25	9															
46	16	0															
47	14	8															
48	03	1															
49	58	2															
50	49	1															
51	41	0															
51	25	0															
Mean level.																	
= 54.5																	

log.....	935.80	= 2.97118
log cos $\delta$ .....	8.98466	
log 15.....	1.17659	
a. c. log 9.....	9.04576	
log R.....	75".804	= 1.87969
refraction.....	—	0.4
on <sup>1</sup> revolution.....	75.7(0	
DIFF. REFRACTION.		
log.....	57".7	= 1.7612
log sin.....	75.8	= 6.5632
log sec Z.....	0.1588	
log sec Z.....	0.1588	
diff. refr.....	—	0.044
log.....	8.6400	



The time from elongation of the star requires a correction in order to reduce the curve in which the star apparently travels to a vertical line. The hour angle of the star is first obtained from the equation,

$$\cos t_o = \cot \delta \tan \varphi,$$

$\delta$  being the star's declination and  $\varphi$  the latitude.

The chronometer time of elongation,  $T_o = \alpha - t_o - \delta t$ ,  $\alpha$  being the right ascension of the star obtained from the Nautical Almanac, and  $\delta t$  the error of the chronometer.

Having thus obtained the chronometric time of elongation, the correction in question is obtained from the observed interval of time of each observation before or after elongation, from tables in Appendix No. 14, United States Coast and Geodetic Survey Report for 1880, pp. 256 and 257. A discussion of this subject will be found in the appendix above referred to and in Chauvenet's Practical Astronomy, vol. 2, pp. 360 to 364.

The times of observation thus corrected for level, and distance from elongation, are then grouped in pairs, selected as being a certain number of revolutions of the micrometer apart, and the time intervals between the members of each pair are obtained. The mean of these, divided by the number of revolutions which separate the members of each pair, is yet to be corrected for differential refraction, which is derived from the equation,

$$\text{refraction} = 57''.7 \sin R \sec^2 Z,$$

$R$  being the value of a division of the micrometer and  $Z$  the zenith distance of the star. Four-place logarithms are sufficient for computing this correction, as it is small. Herewith is an example of record and computation of the value of a revolution of the micrometer of combined instrument No. 534, one of the two in possession of the United States Geological Survey.

#### MEASUREMENT OF A LEVEL DIVISION.

The value of a division of the level is commonly measured with a level trier. The latitude level, however, may be easily tested by means of the micrometer, the value of a revolution being obtained by the following method:

Point the telescope upon some well-defined terrestrial mark and set the level at an extreme reading near one end of the tube. Set the movable thread upon the object and read the micrometer and the level.

Now move the telescope and level until the bubble is near the other end of the tube. Again set the movable thread upon the object and again read both micrometer and level. It is evident that the micrometer and the level have measured the same angle, and that the

ratio between these readings equals that between a revolution of the micrometer and a level division.

An example illustrative of this is appended.

*Determination of value of 1 division of latitude level No. 534.*

[By comparison with micrometer screw 534.]

Micrometer.	Level.		Difference.		aa.	ab.
	N.	S.	Microm.	Level.		
<i>r.</i>	<i>d.</i>	<i>d.</i>	<i>b.</i>	<i>a.</i>		
8.025	47.3	29.2				
8.508	20.7	02.7	48.3	26.55	704.9	1283.
8.509	18.9	01.0				
7.984	49.8	31.0	52.5	30.45	927.2	1599.
8.511	18.5	00.6				
8.045	47.2	29.1	46.6	28.60	818.0	1333.
9.076	18.7	00.8				
8.604	46.0	28.0	47.2	27.25	742.6	1286.
9.442	23.7	05.0				
9.009	48.0	30.0	43.3	24.15	583.2	1046.
10.055	21.8	04.0				
9.574	48.0	30.1	48.1	26.15	683.8	1258.
10.661	24.0	03.1				
10.212	50.7	33.0	44.9	26.80	718.2	1203.
11.771	18.3	00.7				
11.252	48.3	31.9	51.9	30.00	936.4	1588.
12.328	20.0	02.3				
11.872	46.1	28.5	45.6	26.15	683.8	1192.
12.869	22.2	04.6				
12.438	47.7	30.0	43.1	25.45	647.7	1097.
13.468	23.0	05.3				
13.080	44.5	26.9	38.8	21.55	464.4	836.
14.146	20.1	02.4				
13.702	45.4	27.8	44.4	25.35	642.6	1125.
14.758	22.3	04.8				
14.282	48.6	31.0	47.6	26.25	689.1	1249.
Sum					9241.9	16095.

log.....16095. =4.20669.

a. c. log..... 9241.9=6.03424.

log 1 div. micrometer.....=9.87966.

1 div. level.....=1''.320 log.=0.12059.

#### COMPUTATION OF APPARENT DECLINATION OF STARS.

After the determination of the constants of the instrument used, the next step is to obtain the apparent declinations of the stars used. Whenever possible, these should be taken from the Nautical Almanac or the Berliner Jahrbuch. In other cases they must be computed. The positions of stars are given in Safford's Catalog for the epoch 1875.0, together with the annual precession and proper motion. The declinations there given should be revised by the aid of more recent catalogs, particularly with reference to stars of class C. The annual precession and proper motion multiplied by the number of years which have elapsed, and applied, together with the effect of secular variation in precession, give the declination at the beginning

of the year. Further corrections to bring the positions down to the date of observation are expressed by the symbols  $Aa'$ ,  $Bb'$ ,  $Cc'$ ,  $Dd'$ . Logarithms of  $a'$ ,  $b'$ ,  $c'$ ,  $d'$  are given in Safford's Catalog, and  $A$ ,  $B$ ,  $C$ , and  $D$  are given in the Nautical Almanac. A slight additional correction is also to be made for proper motion for the elapsed portion of the year. This reduction is illustrated below:

*Example of reduction. Computation of apparent declination of star 1539.*

[From Safford's Catalog, p. 40.]

Star No. 1539	Declination 1875.0	Annual precession.	Proper motion.
	45 33 29.20	+18.87	-.03

$$\begin{aligned}
 (1890-1875) 15 \times 18''.87 &= +4 \quad 43.05 = \text{Precession for 15 years.} \\
 15 \times -.03 &= -0 \quad 00.45 = \text{Proper motion for 15 years.} \\
 &+0 \quad 00.07 = \text{Secular variation in precession.}
 \end{aligned}$$

$$\begin{aligned}
 45 \quad 33 \quad 11.87 &= \text{Declination 1890.} \\
 +9.38 &= Aa' \\
 -0.78 &= Bb' \\
 +6.88 &= Cc' \\
 +10.16 &= Dd' \\
 -0.03 &= \text{Proper motion, Jan. 1—Nov. 9, 1890.}
 \end{aligned}$$

$$45 \quad 33 \quad 37.48 = \text{Declination Nov. 9, 1890.}$$

$$\begin{array}{cccc}
 \log a' = 1.2757 & \log b' = 9.5264 & \log c' = 9.7367 & \log d' = 9.8273 \\
 \text{Nov. 9. } \log A = 9.6566 & \log B = 0.3649 & \log C = 1.1006 & \log D = 1.1796 \\
 \hline
 0.9723 & 9.8943 & 0.8373 & 1.0066 \\
 Aa' = +9.38 & Bb' = -0.78 & Cc' = +6.88 & Dd' = +10.16
 \end{array}$$

With all this preliminary work done, the reduction proper of latitude observations is comparatively simple. The observations being grouped by pairs, the mean declination of each pair is obtained, the corrections for difference of micrometer readings and levels are applied, with a small correction for differential refraction, and the result is the desired latitude.

Following is an example of the reduction of six pairs of stars observed for latitude at Rapid, S. Dak.:

*Example of reduction.*

[Station: Rapid, S. Dak. November 9, 1890. Half rev. micrometer = 37".900. One div. level = 17.333"]

Date.	Star Nos.	$\delta_1$	$\delta_2$	$\frac{1}{2}(\delta_1 + \delta_2)$	Corrections.			Latitude n.	Weight	
					Microm.	Level.	Refr.			
Nov. 9.	[7 Lacert and 10 Lac. r.]	45 32 87.5	45 29 04.0	45 30 45.75	-1 23.53	-6.51	-0.0344	45 30 01	0.38	3.78
	1539 1551	45 38 37.48	42 41 04.6	44 21 06	-6 31.77	-2.06	-.11	47 12	.90	6.41
	1565 1579	48 33 39.78	49 27 41.04	49 30 41	-6 58.35	-0.56	-.03	52 51	.79	1.98
	1600 1633	56 31 06.66	51 55 56.91	54 04.78	-10 13.25	-3.78	-.19	44 56	.90	4.10
	1676 1686	67 12 10.93	21 03 54.02	44 08.48	-3 08.43	-7.44	-.07	46 54	.93	6.08
	1702 1722	21 32 09.04	63 35 27.51	42 33 43.28	+1 01.51	-5.22	+.02	46 50	.90	5.85
									5.00	30.20

November 9. Weighted mean: 44° 01' 45".59.

## OBSERVATIONS FOR TIME.

With the transit mounted, leveled, and adjusted in the meridian, the chronograph set up and running and connected in a circuit with the battery, and the chronometer and observing key connected in the same circuit, the observer is prepared to begin time observations.

The list of stars which should be used is that given in the *Berliner Jahrbuch*, as the list is fuller and more accurate than that in any other catalog which gives day places. Stars should be so selected north and south of the zenith that the azimuth errors will balance one another as nearly as possible, as is explained hereafter. On the approach of the selected star to the meridian, the telescope should be set by means of the vertical circle upon the altitude of the star above the horizon, deduced from the declination and the latitude. As the star crosses each thread in the reticule, the fact should be recorded by pressing the observing key, which produces, as previously described, a record upon the chronograph sheet. In this way four time stars, as stars between the equator and zenith are designated, and one circumpolar star, or a star so near the pole that it is constantly in sight, should be observed. Then the telescope should be reversed in the wyes and a similar set of stars observed. Between observations upon any two stars the striding level should be placed upon the pivots of the instrument and readings taken to ascertain the departure of the axis from a horizontal position.

In order to avoid unequal expansion of the pivots from unequal heating both bull's-eye lamps must be lighted and placed in their stands.

After the comparison of chronometers at the two stations, to be hereafter described, a similar set of stars should be observed, if possible.

## REDUCTION OF TIME OBSERVATIONS.

Certain constants of the transit should be measured before proceeding with the reduction of time observations. The value of a division of the striding level should be measured by means of a level trier. The equatorial interval of time between each of the threads and the mean of all the threads should be obtained, as it is not infrequently needed in utilizing broken or imperfect observations. These can best be obtained from observations on slow-moving stars, but any stars may be used for the purpose. The intervals as observed are reduced to the equator by multiplying them by the cosine of the declination of the star observed.

The object of these observations is specifically the determination of the error of the chronometer. This error equals the right ascension of a star minus its observed time of transit, corrected for certain instrumental errors. These errors are as follows:

## CORRECTION FOR ERROR OF LEVEL.

The level error, designated by  $b$ , is ascertained from the readings of the striding level. The value of a division of the level in seconds of time must have been previously ascertained by means of a level trier. The effect of the level error is greatest at the zenith and diminishes to zero at the horizon. The correction in seconds of time is given by the equation,

$$\text{cor.} = b \cos (\varphi - \delta) \sec \delta = bB.$$

When the declination is north it is to be regarded as having a plus sign for upper and a minus sign for lower culmination; when south it is negative.

## CORRECTION FOR INEQUALITY OF PIVOTS.

This correction can be made a part of the level correction.

Let  $p$  = the inequality of pivots,

$B$  = inclination of axis given by level for clamp west,

$B'$  = inclination of axis given by level for clamp east,

$b$  = true inclination of axis for clamp west,

$b'$  = true inclination of axis for clamp east;

$$\text{then } p = \frac{B' - B}{4},$$

$b = B + p$  for clamp west,

$b' = B' - p$  for clamp east.

(Chauvenet, vol. 2, p. 155.)

## CORRECTION FOR ERROR OF COLLIMATION.

This correction, designated by  $c$ , is the departure of the mean of the threads from the optical axis of the telescope. For stars at upper culmination, with clamp west it is plus when the mean of the threads is east of the axis, and minus when it is west of it; for stars at lower culmination the reverse is the case. The value of  $c$  is one-half the difference between the clock error indicated by stars observed before and after reversal of the instrument, divided by the mean secant of the declinations of the stars. This is slightly complicated with the azimuth, altho the effect of that is largely eliminated by the proper selection of stars; consequently it is to be obtained by approximations in conjunction with the azimuth errors. The correction to be applied to each star equals  $c \sec \delta - cC$ , which is plus for a star at upper culmination and minus for a star at lower culmination. It is least for equatorial stars and increases with the secant of the declination.

## CORRECTION FOR DEVIATION IN AZIMUTH.

This correction, designated by  $a$ , represents the error in the setting of the instrument in the meridian. Its effect is zero at the zenith and increases toward the horizon. Since the instrument is liable to be disturbed during the operation of reversal, it is necessary to determine the azimuth error, both before and after reversal, separately. A comparison of the clock error, determined from observations upon north and south stars, will furnish the data necessary for the determination of azimuth. Practically, it is determined by elimination from equations involving the mean of all these stars observed in each of the two positions of the instrument, after correcting for level, and as it is slightly complicated with collimation it must be reached by two or more approximations. The error is essentially positive when the telescope points east of south, and negative when west of south. The correction applicable to any star is expressed in the equation,

$$\text{cor.} = a \sin (\varphi - \delta) \sec \delta = aA.$$

It must be understood that the declination when north is positive or upper and negative for lower culmination, and that with south declination it is negative.

## CORRECTION FOR DIURNAL ABERRATION.

The right ascension of stars, as taken from the *Berliner Jahrbuch*, must be corrected for diurnal aberration, which equals  $0''.021 \cos \varphi \sec \delta$ . This correction is positive for upper and negative for lower culmination.

These corrections are summarized in the equation,

$$\delta t = \alpha - (t + aA + bB + cC).$$

A, B, C, as seen above, are constants, depending upon the latitude of the place of observation and the declination of the star. Tables of these quantities will be found in a special publication of the United States Coast and Geodetic Survey for 1874.

## EXAMPLE OF REDUCTION OF TIME OBSERVATIONS.

The following is an example of the form for record of observation and reduction of time observations, taken from a campaign for the determination of the position of Rapid, S. Dak.

Time determination: *Example of record.*

[Rapid, S. Dak., November 20, 1890. Fauth transit, No. 534. Sidereal chronometer: Bond & Sons, No. 187. 1 division of level = 0".118. Hourly rate of chronometer = 0".133.]

Star.....	$\gamma$ Cephei.		$\phi$ Pegasi.		$\alpha$ Piscium.		$\alpha$ Androm.	
Clamp.....	W.		W.		W.		W.	
Level.....	Telescope north. H. Sum. E. 19.8 -88.1 68.3 68.2 +87.6 19.4		Telescope south. H. Sum. E. 68.0 +87.1 19.1 20.2 -89.2 69.0		Telescope south. H. Sum. E. 20.0 -89.5 69.5 68.8 +87.2 18.4		Telescope south. H. Sum. E. 19.8 -89.3 69.5 68.3 +86.8 18.5	
Difference.....	- 0.5		- 2.1		- 2.3		- 2.5	
Thread I.....	h. m. s. 23 34 52.25		h. m. s. 23 47 24.00		h. m. s. 23 54 10.89		h. m. s. 00 03 12.00	
II.....	35 11.40		28.55		14.88		16.83	
III.....	20.41		32.72		19.22		21.94	
IV.....	46.78		36.75		23.14		25.95	
V.....	05.00		41.09		27.20		30.85	
Sum.....	4.84		3.11		5.33		7.00	
Mean.....	23 35 28.97		23 47 32.62		23 54 19.07		00 03 21.40	
Aberration.....	- .07		- .06		- .02		- .02	
Correction for level (b B).....	- .22		+ .06		- .05		- .06	
Correction for rate.....	+ .05		+ .03		+ .01		+ .00	
Reduced transit (I).....	23 35 28.83		23 47 32.57		23 54 19.01		00 03 21.32	
Tabular R. A (a).....	23 34 53.13		46 55.67		53 41.98		00 02 44.42	
a - t.....	- 35.70		- 26.90		- 37.03		- 36.90	

Mean of levels =  $-\frac{2.02}{4} \times .118 = -.0600 = b$ . Inequality of pivots =  $\frac{dir.}{4}$ .

Mean of levels =  $-\frac{2.02}{4} \times .118 = -.0600 = b$ . Inequality of pivots =  $\frac{dir.}{4}$ .

Time determination: Example of record—Continued.

Star.....	$\gamma$ Pegasi.	Br. $\delta$ .	$\alpha$ Ceti.	44 Pleiadem.	12 Ceti.
Clamp.....	E.	E.	E.	E.	E.
Level.....	Telescope south: W. Sum. E. 18.2 -88.3 68.1 68.9 +87.8 18.9 - 0.5	Telescope south: W. Sum. E. 68.7 +87.3 18.6 18.4 -88.7 68.3 - 1.4	Telescope south: W. Sum. E. 19.2 -88.4 68.2 68.5 +86.7 18.2 - 1.7	Telescope north: W. Sum. E. 68.9 +87.8 18.9 18.9 -87.9 68.0 - 0.1	
Difference.....					
Thread V.....					
IV.....					
III.....					
II.....					
I.....					
Sum.....	h. m. s. 00 08 05.25 06.30 13.54 17.65 22.00 7.74	h. m. s. 00 10 05.00 22.81 39.30 56.90 11 15.49 9.50	h. m. s. 00 14 20.70 24.68 28.52 32.90 37.23 4.03	h. m. s. 00 20 17.35 20.84 24.93 29.16 33.42 5.70	h. m. s. 00 24 56.85 25 00.73 05.37 08.16 13.07 5.17
Mean.....	00 08 13.55	39.90	28.81	25.14	05.03
Correction for aberration.....	- .02	- .06	- .02	- .02	- .02
Correction for level ( $\delta$ B).....	- .02	- .09	- .02	- .02	- .02
Correction for rate.....	- .02	- .02	- .03	- .04	- .05
Reduced transit ( $t$ ).....	00 08 13.49	00 10 39.73	00 14 28.74	00 20 25.06	00 25 04.94
Tabular R. A. ( $\alpha$ ).....	00 07 38.59	00 10 03.56	00 13 51.75	00 19 48.17	00 24 27.91
$\alpha-t$ .....	-38.90	-38.17	-38.99	-38.89	-37.03

div.  
 $\frac{.925}{4} \times .118 = -.027 - \delta$ . Inequality of pivots = .00.  
 Mean of levels =  $-.027 - \delta$ .



## Example of reduction.

[Rapid, S. Dak., November 20, 1880. After exchange of clock signals.]

Clamp.	1.	2.	Star.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12.	13.	V.	t.
				A.	C.	a-t	Cc.	Corrected for Cc.	Aa.	Corrected for Aa.	Cc.		As'w.			
W.	W.T.						$c = -.019$				$c' = +.015$					
	1	γ Cephei.....		-2.42	+4.45	-35.70	-1.08	-33.78	-1.00	-36.78	+0.07	-36.71	-0.05	-36.76	8.00	23.58
	2	γ Pegasi.....		+0.45	1.05	-36.80	-0.02	-36.82	+0.19	-36.73	+0.01	-36.73	+0.01	-36.71	-0.03	.78
	3	ω Piscum.....		+0.62	1.01	-37.03	-0.02	-37.05	+0.26	-36.79	+0.02	-36.77	+0.01	-36.76	.00	.89
	4	β Piscum.....		+0.77	1.01	-37.13	-0.02	-37.15	+0.32	-36.83	+0.01	-36.82	+0.02	-36.80	+0.04	.99
	5	α Androm.....		+0.30	+1.14	-36.90	-0.02	-36.92	+0.12	-36.80	+0.02	-36.78	+0.01	-36.77	+0.01	24.01
		Sum.....		+2.14	+8.66	-183.66				3.93				3.80		
	(1)	Mean.....		-2.42	+1.73c	-36.732				-36.786				-36.760		
E.	1	γ Pegasi.....		+0.51	-1.03	-36.90	+0.02	-36.88	+0.15	-36.73	-0.01	-36.74	-0.01	-36.75	-0.01	24.13
	2	β Androm.....		-2.26	4.23	-36.17	.06	-36.09	-0.64	-36.73	.06	-36.79	+0.03	-36.76	.00	.17
	3	γ Cephei.....		+0.81	1.01	-36.99	.02	-36.97	+0.23	-36.74	.02	-36.76	-0.01	-36.77	+0.01	.23
	4	ω Piscum.....		+0.68	1.00	-36.80	.02	-36.87	+0.19	-36.68	.01	-36.69	-0.01	-36.70	+0.06	.33
	5	α Androm.....		+0.75	-1.00	-37.03	+0.02	-37.01	+0.21	-36.80	-0.02	-36.82	-0.01	-36.83	+0.07	.41
		Sum.....		+2.75	-8.27	-183.98				3.68				3.81		
	(2)	Mean.....		-2.26	-1.65c	-36.796				-36.736				-36.762	Mean	-24.05

Subtracting (2) from (1), ignoring azimuth terms which are small, we have:

$$\begin{array}{r} \text{Approx } \dots c = -.019 \\ \text{From below } c' = +.015, \text{ Adopted } \dots c = -.004 \\ \hline -2.42 \text{ Aw} - 35.78 = 0 \quad \delta_s \\ +0.54 \quad -37.01 = 0 \quad +0.69 \quad -36.93 = 0 \end{array}$$

$$+2.96 \text{ Aw} - 1.23 = 0 \quad \delta_s \quad +2.95 \text{ Ae} - 0.94 = 0 \quad \delta_s$$

$$\text{Aw} = +.415 \quad \text{Ae} = +.285$$

Forming equations to determine  $c'$  from columns 4 and 9:

$$\begin{array}{r} +1.73c' - 36.78 = 0 \\ -1.65 \quad -36.73 = 0 \\ \hline +3.38c' - 0.050 = 0 \\ \delta_s \\ c' = +.015 \end{array}$$

Forming equations from columns 3 and 11.

$$\begin{array}{r} -2.42 \text{ Aw} - 36.710 = 0 \quad \delta_s \\ +0.54 \text{ Aw} - 36.772 = 0 \quad \delta_s \end{array}$$

$$+2.96 \text{ Aw} - 0.962 = 0 \quad \delta_s \quad +2.95 \text{ Ae} + 0.038 = 0 \quad \delta_s$$

$$\begin{array}{r} \text{Aw} = +.021 \quad \text{Ae} = -.013 \\ \text{Aw} = +.415 \quad \text{Ae} = +.285 \end{array}$$

$$\text{Adopted } \text{Aw} = +.436 \quad \text{Adopted } \text{Ae} = +.272$$

Adopted  $\dots C = .004$   
Normal equation, formed from columns 1, 3, 4, and 5

$$108t - .28 \text{ Aw} + .49 \text{ Ae} + .39c + 367.64 = 0 \quad \delta_s$$

$$\delta t - .028 \text{ Aw} + .049 \text{ Ae} + .039c + 36.764 = 0 \quad \delta_s$$

$$\delta t = -36.76 \quad \text{at } 24.05$$

$$\begin{array}{r} +.039 \times +.004 = -.000 \\ \delta t = -36.763 \end{array}$$

$$a - t = -36.764 \quad \delta_s$$

$$.028 \times +.436 = -.012 \quad \delta_s$$

$$+.049 \times +.272 = +.013 \quad \delta_s$$

## COMPARISON OF TIME.

After time has been thus observed the chronometers at the two stations should be compared by telegraph.

Chronometers are compared in the following manner: The chronometer at one station being in circuit with the chronograph and recording upon it, the chronometer at the other station is switched into the general telegraphic circuit, by which it is brought to the first station and switched into the local circuit there, so that the two chronometers register upon the same chronograph, their beats being marked side by side by the same pen. After this has gone on for a minute or more the operation is reversed, the chronometer at the first station being switched into the telegraphic circuit and made to record upon the chronograph with the chronometer at the second station. Of course the observers are informed of the hour and minute at which the joint record upon the several chronographs begins.

This method constitutes what is known as the *automatic* exchange of signals.

The *arbitrary* exchange of signals is made as follows:

Each chronometer recording on its own chronograph as usual, and each local circuit being connected with the main-line circuit, the observer at one station breaks the circuit by means of the main-line talking key, which break is recorded on the chronograph sheets at both stations. The breaks are repeated every two seconds for at least one full minute. The operation is then reversed by the observer at the second station making the breaks, which are recorded at both stations as before.

The differences of time between the chronometers at the two stations are read from the chronograph sheets at each station and corrected for error of the chronometers. The results from the two chronograph sheets differ by an amount equal to twice the time occupied in transmission of signals. The mean of the two is therefore the approximate difference of longitude.

This result is yet to be corrected for personal equation, or the difference between the errors of observing of the two observers. Every observer has the habit of recording a transit a little too early or too late, the difference between two observers not infrequently being as great as a fourth of a second. To measure this difference, the observers usually meet, preferably at the known station, both before and after the campaign, and observe for a time each with his own instrument or with one similar in all respects to that used in the campaign. A comparison of the time determinations made by the two observers gives an approximation to the personal equation.

A better method, but one not always practicable, is for the observers, having completed half of the observations for time and longitude, to

exchange stations for the remainder of the work. The mean of the results before and after exchange of stations will eliminate personal equation.

There is one error incident to this work which can not be eliminated. This is the unequal attraction of gravity, or local attraction, or, as it is sometimes called, station error. A neighboring mountain mass will attract the plumb line and deflect the spirit level to such an extent as to cause serious errors in astronomic determinations of latitude and time. The same result is frequently produced by a difference in density of the underlying strata of rock, so that station errors of magnitude often appear where they are not expected. Indeed, the station error can not be predicted with any certainty as to amount or direction.

The only practical method of even partially eliminating this error is to select a number of stations for astronomic location under conditions as widely diverse as possible, connect them by triangulation, and by this means reduce all these astronomic determinations to one point, thus obtaining for this point a number of determinations each having a different station error. The mean of these gives for this point a position from which—in part, at least—station error has been eliminated, and this mean position can be transferred back to the several astronomic stations by means of the triangulation, thus giving each of them a position similarly comparatively free from station error.

#### OBSERVATIONS FOR AZIMUTH.

The initial direction from which the directions of other lines in primary triangulation and in primary traversing are computed is obtained by means of astronomic observations. Such observations should be taken not only upon the initial line, but at intervals thruout the work for its verification. Such intervals should not exceed 100 miles in the primary triangulation and 10 to 20 miles in primary traversing.

Azimuth observations are made with the theodolite used in primary triangulation or traverse. The observations consist in the measurement of the horizontal angle between some close circumpolar star, usually Polaris, and a terrestrial mark, generally a lantern set at a distance of half a mile to a mile from the observing station. The time of observation on the star should be noted by a chronometer or a good watch. As the star is at a much higher angle of elevation than the lamp, it is necessary not only to level the instrument carefully but to measure the error of level and to correct for it. It is therefore essential that the value of a division of the level bulb be known. These observations for azimuth may be made at any time

of the night, but it is very important that they be made at or near the time of elongation of the star, as it is then moving most slowly in azimuth, and any error in the time of observation has the least effect on the resulting azimuth. If such observations be taken at elongation, the reduction of the observations is simplified. When such observations are made at any other time than at elongation, the time must be noted, as it forms an element in the reduction. The error of the clock or watch used can be obtained by comparison with standard time, making correction for the difference in longitude between the station and the meridian of the standard time. A form of observation and record is appended.

*Example of record of azimuth observations.*

[Station: West base, near Little Rock, Ark. Fauth 8" theod. No. 300. December 27, 1888. 1 div.  
micr.=2". 1 div. level=3".]

Object.	Time P. M.			Level.		Micrometer.			Mean.	Angle.
				West end.	East end.	A.	B.			
						Telescope direct.				
	<i>h.</i>	<i>m.</i>	<i>s.</i>	<i>Dir.</i>	<i>Dir.</i>	<i>°</i>	<i>'</i>	<i>Dir.</i>	<i>°</i>	<i>'</i>
Polaris.....	11	00	18	13.9	47.1	346	00	14.8	165	58 25.1
				50.5	10.2				345	59 39.9
				64.4	57.3					
				+7.1						115 32 30.0
East base (mark).....						101	32	18.1	281	31 21.8
East base (mark).....						101	32	19.8	281	31 19.7
Polaris.....	11	09	20	50.4	10.3	345	58	22.0	165	57 01.4
				13.8	46.5				345	57 53.4
				64.2	56.8					
				+7.4						
						Telescope reverse.				
Polaris.....	11	17	14	50.5	10.1	211	28	29.0	31	27 23.4
				12.9	46.6				211	28 22.4
				63.4	56.7					
				+6.7						115 35 53.8
East base (mark).....						327	05	06.7	147	03 09.5
East base (mark).....						327	04	26.3	147	03 00.6
Polaris.....	11	26	22	14.3	46.3	211	27	10.7	31	26 07.4
				50.1	10.5				211	26 48.1
				64.4	56.8					
				+7.6						

*Summary of results of azimuth observations.*

[Station: West base, Arkansas. December 27, 1888.]

Individual results.				Combined results.			
First set.....	294 10	34.2)	35.25 D.	}		38.80	
		30.3)					
		49.9)	42.35 R.	}			
		34.8)					
		35.9)	41.10 R.	}		39.38	
		46.3)					
Second set.....		41.8)	37.65 D.	}			
		33.5)					
		42.4)	43.90 D.	}		38.75	
		45.4)					
		26.4)	33.60 R.	}			
		40.8)					
		49.1)	47.05 R.	}		40.10	
		45.0)					
Grand mean.....		40.3)	33.15 D.	}			
		26.0)					
				294 10	39.26		

## REDUCTION OF OBSERVATIONS FOR AZIMUTH.

The time of observation of a star should be first corrected for the difference in longitude, assuming that standard time has been used, and for the error of the watch. It is then reduced from mean to sidereal time. From the sidereal time of observation should be subtracted the right ascension of Polaris, if that star is used, which is given in the Nautical Almanac, the result being the hour angle or the sidereal time which has elapsed since it past the meridian of the place of observation, given in hours, minutes, and seconds. This result should be converted into degrees, minutes, and seconds.

$$\text{Then } \tan A = -\frac{a \sin t}{1 - b \cos t};$$

where  $a = \sec \varphi \cot \delta$  ( $\varphi$  = the latitude),

$$b = \frac{\tan \varphi}{\tan \delta} (\delta = \text{the declination of star}),$$

$t$  = hour angle,

and  $A$  = angle between true north and the star.

This angle should be corrected for level as follows:

$$\text{level correction} = -\frac{d}{4}\{w+w'\} - (e+e')\}\tan h;$$

$d$  being the value of a division of the level;

$w+w'$ , readings of west end of level bubble;

$e+e'$ , readings of east end of level bubble;

$h$ , the angular elevation of pole star.

An example of reduction is as follows:

*Example of reduction of azimuth observations.*

[Station: West base; December 27, 1888. Observer, S. S. G. Latitude= $34^{\circ} 45' 26.8''$  Longitude  $92^{\circ} 13' 31.5''$ ]

Time of observation	$T^w=11$	$00$	$18$
Correction; ninetyeth meridian time to $92^{\circ}.215$	$-$	$8$	$54$
Watch slow; ninetyeth meridian time	$+$		$02$
Local mean time	$T^m=10$	$51$	$26$
Correction; mean to sidereal time	$-$	$+1$	$47$
Right ascension mean sun		$18$	$26$
		$36$	
Sidereal time of observation		$=29$	$19$
R. A. Polaris		$-1$	$18$
		$25$	
Hour angle	$t=28$	$01$	$24$
	$-24$		
	$h.$	$m.$	$s.$
$t$ (time)	$=4$	$01$	$24$
	$^{\circ}$	$'$	$''$
$t$ (arc)	$=60$	$21$	$00$

$$\tan A = -\frac{a \sin t}{1 - b \cos t} \text{ where } a = \sec \phi \cot \delta \text{ and } b = \frac{\tan \phi}{\tan \delta}$$

$\phi=34$	$45$	$26.8$	$\log \sec = 0.0853539$	$\log \tan .34$	$45$	$26.8=9.8413076$
$\delta=88$	$43$	$11.9$	$\log \cot .88$	$43$	$11.9=1.6508310$	
$\log a$			$=8.4345229$	$\log b$		$=8.1904766$
$\log \sin t$	$60$	$21$	$00$	$\log \cos t$		$=9.6943423$
$\log a \sin t$			$=8.3735744$	$\log -$	$.0076704$	$=7.8848189$
$\log (1-b \cos t)$			$=9.9966559$	$+$	$1.0000000$	
$\log \tan A$	$178$	$38$	$08.0$	$=8.3769185$	$0.9923296$	$=1-b \cos t$
angle to	$+115$	$32$	$30.0$			
mark						
Level correction			$-3.8$	level corr.	$= -\frac{d}{4}\{w+w'\} - (e+e')\}\tan h.$	
Az. of mark	$-294$	$10$	$34.2$	$- \frac{3.1}{4}$	Div.	
					$\times 7.1 \times .694 = -3.8$	

When observations for azimuth are to be made at elongation, it is necessary to know the mean time of elongation. This is computed by the following method:

The hour angle at elongation is obtained from the equation,

$$\cos t = \tan \phi \cot \delta.$$

The hour angle plus the right ascension of the star gives the sidereal time of its western elongation, which, reduced to mean time, gives the local mean time in question.

The azimuth of a pole star at elongation is determined by the use of the equation,

$$\sin A = \sec \varphi \cos \delta.$$

The following is an example of these computations:

*Example of the computation of the azimuth at elongation, and the local mean times of both elongations of Polaris.*

[Latitude  $= \phi = 40^\circ$ . Meridian of Washington. November 28, 1891.]

Sine azimuth at elongation $= \sec \phi \cos \delta$ .	
log sec $40^\circ$	0.1157460
log cos $\delta$	88 44 05.5 $-8.3439803$
log sin A	1 39 05.8 $-8.4597263$
Cos hour angle at elongation, $t_e = \tan \phi \cot \delta$ .	
log tan $40^\circ$	0.9238135
log cot $\delta$	88 44 05.5 $-8.3440862$
log cos $t_e$	88 56 17.5 $-8.2678997$
	<i>h. m. s.</i>
	$t_e = 5 \quad 55 \quad 45.2$
Sidereal time, western elongation, $T_e = \text{R. A. Polaris} + t_e$ .	
R. A. Polaris	<i>h. m. s.</i>
$t_e$	$= 1 \quad 19 \quad 35.2$
	$= 5 \quad 55 \quad 45.2$
Sidereal time, western elongation, $T_e =$	7 15 20.4
R. A. mean sun $=$	$a_s = 16 \quad 29 \quad 14.4$
Sidereal interval before noon,	$- 9 \quad 13 \quad 54.0$
Correction sidereal to mean interval $=$	1 30.7
Mean interval before noon	$= 9 \quad 12 \quad 23.3$
Local mean time, western elongation $=$	2 47 36.7 Nov. 28.
	<i>h. m. s.</i>
Sidereal time, eastern elongation $= 24^h + a - t_e =$	19 23 50.0
	$a_s = 16 \quad 29 \quad 14.4$
Sidereal interval after noon,	$= 2 \quad 54 \quad 35.6$
Correction sidereal to mean interval	$= 0 \quad 28.6$
Local mean time, eastern elongation	$= 2 \quad 54 \quad 07.0$
Local mean time, western elongation	$= 2 \quad 47 \quad 36.7$

For longitudes west of Washington, decrease times of elongation 0.66 second for each degree.



## CHAPTER IV.

### BASE LINE, PRIMARY CONTROL, AND ELEVATIONS.

The primary control or geometric work is, in the ordinary case, effected by triangulation. Wherever this is not practicable or not economical, resort is had to what is known as primary traversing, but wherever the country presents sufficient relief for the purpose triangulation is employed, as it is more accurate and cheaper. In some parts of the country triangulation of sufficiently accurate character for controlling the topographic map of the United States has been executed by other organizations, notably by the Coast and Geodetic Survey and the Lake Survey. Wherever such triangulation is available the results should be utilized.

### PARTY ORGANIZATION.

The primary triangulation is generally carried on by a special party. Under certain circumstances, however, it is economical and advisable for one party to do all the work. The disadvantage is that it divides the time and attention of the topographer, requiring him to turn from one thing to another; the advantage, that it insures the selection of such points as are needed by the topographer. If the work is done by a special party, the points selected are more likely to be chosen on account of their forming good figures in the triangulation than on account of their convenience and usefulness to the topographer. The secondary triangulation, the traversing, and the sketching should be carried on by different men, but under a single party organization. The sketching should be done by the chief of party, the secondary triangulation and height measurement by his most experienced assistant, and the traversing, with height measurement, by the other assistants.

### BASE-LINE MEASUREMENT.

#### SITE.

The measurement of the base line is ordinarily the first of the preparatory steps toward map making. Upon the proper selection of its site and its correct measurement depends all the subsequent work of triangulation. The site must be reasonably level. It is not essential that it be absolutely so, but the more closely it approaches a plane

the less difficulty will be experienced in making an accurate measurement. The site should afford sufficient room for the measurement of a base about 5 miles in length. A base much less than 5 miles in length is not always an economical one, inasmuch as it is less costly to extend the base than to complicate the expansion. A much greater length than 5 miles is unnecessary, because this length permits of easy expansion, and if the length be greater than this it may be difficult to construct intervisible signals at the ends of the base.

The ends of the base must be intervisible, and they must be so situated with regard to suitable points for expansion and triangulation as to form well-proportioned figures. Whenever possible, the base line should form a side or diagonal of a closed quadrilateral or pentagonal figure.

While it is unnecessary to devote time to obtaining extreme accuracy in the measurement of a base, this measurement should be so nearly accurate that its errors can not affect the map altho multiplied many times in the associated triangulation. All necessary precaution should be taken to secure this result.

#### METHODS AND INSTRUMENTS USED.

Various methods and instruments have been employed in the measurement of base lines by the United States Geological Survey. At first wooden rods were employed, varnished and tipped with metal. When used in measuring, these were supported upon trestles and contacts were made between them with considerable refinement. The advantage of using these rods consisted in the fact that their length was but slightly affected by temperature, which is the main source of error in base-line measurement, and being thoroly varnished they were not greatly affected by moisture.

Subsequently bars of metal were employed of the pattern known as the Coast Survey secondary bars. These consisted each of a steel rod between two zinc tubes. As the two metals expand at different rates under changes of temperature, their relative lengths at any temperature as compared to the relative lengths at a normal temperature was theoretically an indication of the temperature of the bars at any time. The arrangement for indicating their relative lengths formed part of the apparatus, and was intended to indicate the temperature of the bars, and thus to afford means of reducing their lengths to a normal temperature. These bars have not been found, however, to work well in practise. Besides, there are other objections to the use of bars of any kind, which may be summarized as follows: First, their use is expensive; a considerable number of men are needed, and as the measurement proceeds slowly it often requires from a month to six weeks to measure and remeasure a base 5 miles in length. Again,

since these bars are but 4 to 6 meters in length, there are many contacts to be made in each mile of measurement, and each contact offers the possibility of a slight error.

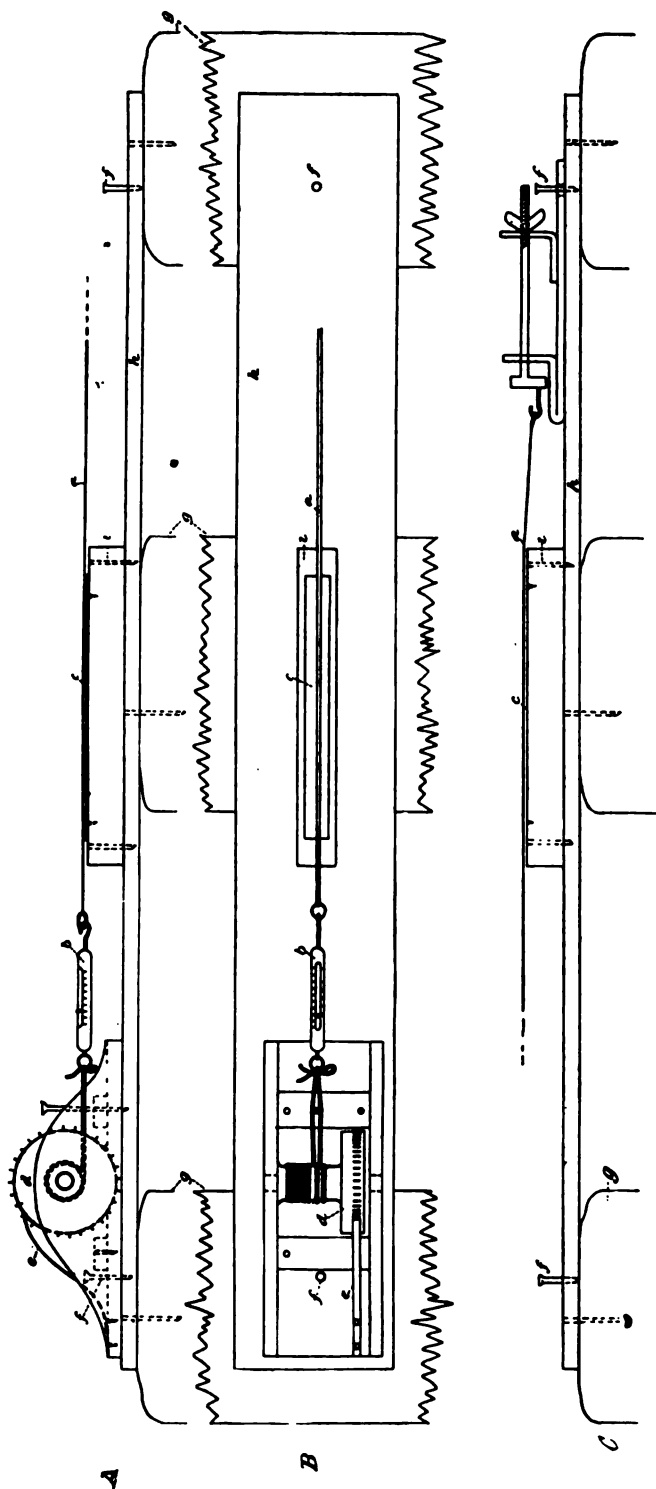
In view of these objections and of certain positive advantages which the change would produce, it was decided to drop the use of bars in the measurement of base lines and to adopt long steel tapes. By their use it has been found easy to attain the required degree of accuracy in measurement, inasmuch as the number of contacts is much smaller than when bars are used, while the uncertainty in regard to the temperature of the measuring apparatus is reduced to a minimum by carrying on the measurement at night or in cloudy weather. The expense of the measurement is greatly reduced, since fewer men are required, the work of preparing the ground and the work of measuring are much lessened, and the rapidity of measuring is increased manyfold. The diminished cost makes it practicable to measure much longer bases, thus decreasing the number of stations required in the expansion. This method allows, also, the measurement of base lines at shorter intervals in the triangulation.

The tape in use has a length of 300 feet. It should be carefully compared, at an observed temperature, with a standard, both before and after its use in base measurement. Preferably, the site for the base line should be selected along a railway tangent, as such location is approximately level, and the railway ties afford an excellent support for the tape. If such a location is not available, one should be selected that will fill the requirements previously mentioned, should be cleared of brush and undergrowth, and if necessary its sharp inequalities should be leveled. The tape should be supported at intervals of not more than 25 feet by a series of low stools whose legs are prest into the ground, or by a series of laths driven into the ground, each bearing a horizontal support made of a wire nail, while similar stools or laths should sustain each end of the tape.

The measurement of a base with the steel tape is a simple matter. Provision must, however, be made, first, for the proper alignment of the base; second, for the proper tension of the tape; and, third, for the measurement of temperature.

The alignment also is simple, and should be marked upon the ground in advance of the work of measurement. In cases where a railway tangent furnishes the site for the base line, no alignment is needed beyond the provision for keeping the tape always at a uniform distance from one of the rails.

For insuring a uniform tension of the tape, an ordinary spring balance should be attached to the forward end of the tape, where a tension of 20 pounds is applied. In order to apply this uniformly and to insure against slip of the tape, an apparatus has been devised by Mr. H. L. Baldwin, jr., of the U. S. Geological Survey. It is



A. Forward end, profile  
B. " " plan  
C. Rear " profile

a. Tape b. Spring balance c. Zinc strips d. Cog wheel for straining tape e. Ratchet f. Nail for holding gear in place g. Railroad tie h. Pine strip i. Block support for sunstrip

DEVICE FOR STRETCHING TAPE IN BASE MEASUREMENT.

Designed and used by H. L. Baldwin, Jr.



shown in Pl. II. For its use, it is necessary to obtain strips of board about 5 feet long and 4 inches wide, in number equal to the number of lengths of tape of which the base line consists. Numbered strips of zinc, each about 8 inches long and 1 inch wide, are tacked to the boards, and the boards are nailed to the railway ties if measurement is to be made along a railway tangent. These boards are designed to support the devices for maintaining the tension, and the contacts are marked upon the strips of zinc. Mr. Baldwin's apparatus consists essentially of a wheel worked by a lever and held by ratchets in any desired position. This wheel is attached to the spring balance in such way that by turning it the strain is put upon the spring balance, which is held at the desired tension by the ratchets. A small mechanism at the rear end of the tape holds the zero of the tape at the opposite mark. The great length of the tape, 300 feet, allows considerable friction or drag when the supports are frequent, and in order to insure a reasonably uniform distribution of the strain upon the tape it should be raised and allowed to fall with the strain on.

The measurements should be made at night or during cloudy days, in order that the temperature of the air, which is that indicated by the thermometers, and that of the tape shall be as nearly as possible the same. The temperature must be carefully observed by at least two thermometers at each tape length, in order that the best possible data for temperature correction may be obtained.

The base should be measured at least twice, and the two results compared by sections of 1,200 feet, or four tape lengths. The ends of the base should, if possible, be permanently marked by means of stone monuments set into the ground so that their surfaces are but a few inches above ground level, and the exact position of the ends should be indicated by a cross cut in a copper bolt embedded in the head of the stone, in order that the base may be preserved for future references.

A line of levels must be run over the site or over the stools which support the tape, for the purpose of obtaining its profile and thereby the means for deducing its horizontal length.

#### PERSONNEL OF PARTY.

The personnel required in the measurement of a base line should be, in an ordinary case, as follows:

The chief of the party, who exercises a general supervision over the work, marks the extremities of the tape, and provides the necessary precautions against errors in the measurement, as hereafter stated.

The rear tapeman, who adjusts the rear end of the tape to the contact marks and who carries and reads one of the thermometers.

The head tapeman, who adjusts the forward end of the tape, exerts the requisite tension upon it, and carries and reads a second thermometer.

A recorder.

### REDUCTION OF BASE-LINE MEASUREMENT.

#### REDUCTION TO STANDARD.

The first correction to be applied is that of reduction to a standard. The correction for this should be obtained by comparison with the standard of the Bureau of Standards. The correction for the entire line is in proportion to the correction as obtained by comparison with the standard. If the tape be longer than the standard the correction will be positive; if shorter, negative.

#### CORRECTION FOR INCLINATION.

Data are obtained by running a line of levels over the base line. This line of levels gives the rise or fall, in feet and decimals of a foot, between the points of change in inclination. From this and the measured distance the angle of inclination is computed from the formula,  $\sin \theta = \frac{h}{R}$ ;  $R$  being the distance and  $h$  the difference in height, both given in feet. The correction in feet to the distance is then computed by the equation,

correction =  $\frac{\sin^2 \frac{1'}{2} \theta^2 R}{2}$  or  $0.00000004231 \theta^2 R$ ,  $\theta$  being expressed in minutes. (See Lee's Tables, p. 83.)

#### CORRECTION FOR TEMPERATURE.

Steel expands for each degree of temperature .0000063596 of its length. This fraction multiplied by the average number of degrees of temperature at the time the base line was measured above or below 62 degrees, which is taken as the normal temperature, gives the proportion in which the base line is to be diminished or extended on account of this factor. Care must be taken to obtain correctly this average temperature. It must be the mean of all the thermometric readings, taken at uniform intervals of distance during the measurement. If the temperature be above the normal the correction is positive, and vice versa.

#### REDUCTION TO SEA LEVEL.

The base line is measured on an arc of a circle parallel to the sea surface and raised above it, at an elevation which is known at least approximately. This arc with radii drawn from its extremities to

the center of the earth forms approximately a triangle similar to that formed by the radii of the earth with the sea surface. The length at sea level is derived with a sufficient approximation to correctness by the proportion,

$$R : h :: K : \text{correction,}$$

R being the radius of the earth,  $h$  the mean height of the base line above sea level, and K its measured length. (See Report United States Coast and Geodetic Survey, 1882, Appendix 9. p. 196.)

#### EXAMPLE OF REDUCTION OF BASE-LINE MEASUREMENT.

The following is an example taken from the records of measurement of a base near Spearville, Kans., together with the reduction of this base for inclination, temperature, and elevation above sea level:

##### *Record of measurement and reduction of Spearville base, Kansas.*

[Section 1. Stations 0-10. October 16, 1889. Light rain falling.]

No. of tape.	Time.		Tension.	Thermometers.		Temperature correction.	Total length of section.
	a. m.			A.	B.		
	<i>h.</i>	<i>m.</i>	<i>Lbs.</i>	°	°		
1.....	10	13	19.75	50.5	50.0	Mean temp. = 50.51	
2.....		20	20.00	50.5	50.0		
3.....		26	20.00	50.5	50.0		
4.....		31	20.25	50.5	50.0	° ° °	
5.....		37	20.00	50.7	50.5	62 50.51 = 11.49	
6.....		42	20.125	51.5	50.6	°	
7.....		47	20.25	51.0	50.8	-11.49 × 3000.	1 tape length.....
8.....		51	20.00	50.8	50.2	× .000006	Feet. = 300.0617
9.....		55	20.125	50.8	50.0		10 × 300.0617.....
10.....		58	20.00	50.7	50.5	= .207 foot.	Temperature correction.....
							Result first measurement.....

[Second measurement, October 17, 1889.]

No. of tape.	Time.		Tension.	Thermometers.		Temperature correction.	Total length of section.
	p. m.			A.	B.		
	<i>h.</i>	<i>m.</i>	<i>Lbs.</i>	°	°		
1.....	12	13	20.00	52.3	52.4	Mean = 53.96	
2.....		21	20.25	53.3	52.9		
3.....		25	20.00	53.8	54.0		
4.....		29	19.75	55.0	54.8	° ° °	
5.....		33	20.00	55.0	53.2	62 53.96 = 8.04	
6.....		36	20.00	53.8	54.0	°	
7.....		38	20.00	54.0	54.0	-8.04 × 3000.	
8.....		41	20.12	54.5	54.0	× .000006	10 × 300.0617.....
9.....		45	19.75	55.1	54.4		Feet. = 3,000.617
10.....		50	20.13	54.5	54.1	= .145 foot.	Set back.....
							Temperature correction.....
							Result second measurement.....



*Correction for inclination, Spearville base, Kansas.*

$$\text{Correction} = \frac{\sin^2 1'}{2} \theta^2 \times \text{Distance.}$$

Approximate distance.	Difference of elevation.	Angle $\theta$	Log $\theta$	2 log $\theta$	Log $\frac{\sin^2 1'}{2}$	Log dist.	Log correction.	Correction.
<i>Feet.</i>	<i>Feet.</i>	<i>' "</i>						
200	0.8	13 34	1.1326	2.2652	Constant.	2.3010	7.1926	.0015
4,200	4.2	2 22	0.3674	0.7348		3.6232	6.9844	.0010
4,000	12.0	10 08	1.0052	2.0104		3.6021	8.2389	.0173
1,000	1.0	3 23	0.5250	1.0501		3.0000	6.6765	.0005
2,000	3.0	5 04	0.7024	1.4049		3.3010	7.3323	.0021
4,200	22.0	12 23	1.0917	2.1834		3.6232	8.4330	.0271
2,800	7.0	8 27	0.9263	1.8527		3.4472	7.9263	.0084
1,000	0.0	0 00	0.0000	0.0000		3.0000	0.0000	.0000
1,000	1.0	3 23	0.5250	1.0500		3.0000	6.6764	.0005
4,200	20.0	11 16	1.0504	2.1008		3.6232	8.3504	.0224
3,800	6.0	5 20	0.7267	1.4535		3.5798	7.6597	.0046
2,000	4.0	6 45	0.8293	1.6586		3.3010	7.5860	.0038
5,400	31.4	19 39	1.2934	2.5867		3.7324	8.9455	.0882
2,000	2.6	4 24	0.6437	1.2874		3.3010	7.2148	.0016
135	0.05	1 18	0.1072	0.2144		2.1303	4.9712	.0000
								.1790

*Reduction to sea level.*

Correction.....	K A
	R
Log K (meters).....	-4.05656
Log h (meters).....	-2.87599
Colog R.....	-3.19660
Log 1.356 meters.....	-0.13215
Log meters to feet.....	-0.51599
Log 4.448 feet.....	-0.64814

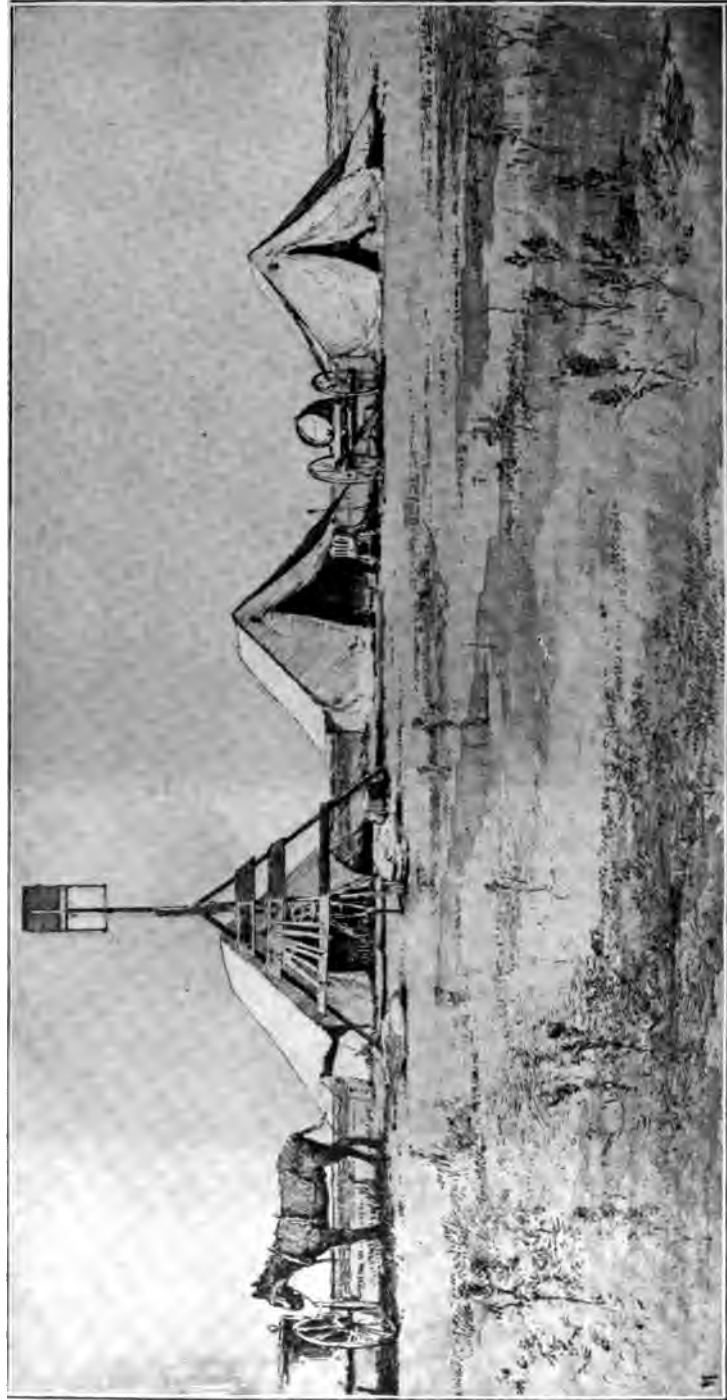
*Spearville base: Summary by sections.**[Corrected for temperature.]*

Stations.	First measure.	Second measure.	Difference.
			<i>First - Second.</i>
1 to 10	3,000.410	3,000.401	+ .009
10 20	.418	.393	+ .025
20 30	.431	.431	+ .000
30 40	.426	.446	-.020
40 50	.437	.478	-.041
50 60	.417	.455	-.038
60 70	.369	.392	-.023
70 80	.366	.356	+ .010
80 90	.955	.938	+ .017
90 100	.676	.667	+ .009
100 110	3,000.899	3,000.898	+ .001
110 119	2,700.581	2,700.571	+ .010
119 126	2,100.244	2,100.234	+ .010
	37,806.629	37,806.660	-.031 = .372

Mean of 2 measurements.....	-37,806.645
Reduction from S. W. base to $\Delta$ .....	-168.235
Reduction from N. E. base to $\Delta$ .....	-2.864
Correction for inclination.....	-0.179
Reduction to sea level.....	-4.448
Corrected length.....	37,630.919

*a Corrected for temperature.*





FRAMED SIGNAL FOR USE WHERE OBSERVING TOWER IS NOT NECESSARY.

## PRIMARY TRIANGULATION.

The base line having been measured, the next step is the expansion. This work, as well as the body of the triangulation, consists in the selection of stations, the erection of signals, and the measurement of angles. Each triangle from the base line outward will, when the angular measurement is completed, have one side and the three angles known, from which the other two sides can be computed by means of a simple trigonometric formula.

The expansion differs from the body of the triangulation only in the fact that the average length of the sides of the triangles is less. As the expansion progresses away from the base line the sides of successive triangles become gradually longer, until the average length of side of the triangulation is reached. Since the sides are increasing in length, and hence since any inaccuracy in the measurement of the base is multiplied, this work must be planned and executed with greater care than the body of the triangulation requires.

A base line measured as above prescribed requires little expansion, since from the extremities of a 5-mile base one can observe directly on points 8 to 10 miles away, a distance as great as the average side of a triangle. Ordinarily, from the ends of the base, the surveyor can observe directly upon stations in his scheme of triangulation.

In the Western mountain region of the United States, where the sides of triangles may be 20 to 50 miles in length, an expansion is required.

## SELECTION OF STATIONS.

In the selection of triangulation stations two sets of requirements must be observed.

(1) Such stations must be so selected as to afford what is known as strong figures, in order to reduce to a minimum the errors which will creep into an extended system. In order to insure intervisibility, they should, if possible, be located upon hill or mountain summits, the most commanding in the neighborhood. No triangle on which dependence is placed for the location of a station should have at that station an angle of less than 30 degrees or more than 150 degrees.

The stations should, if practicable, be grouped into simple figures, as quadrilaterals or pentagons, with an interior station, etc. Where an area is being covered with triangulation such groupings naturally occur, but in certain cases the triangulation takes the form of narrow belts of figures, and then the belt may consist of simple triangles or quadrilaterals, as more complex figures are rarely desirable.

(2) Since the sole object of triangulation is the control of the topographic map, the location of stations should be adjusted to the needs of the topographers as far as is consistent with accuracy. This requirement affects most seriously the distance between stations.

Every plane-table sheet should contain at least three primary stations, and a fourth is desirable. Thus, for controlling the sheets on the scale 1:62500, the stations should not be more than 10 or 12 miles apart, and should be located with direct reference to the control of certain sheets. Again, since the primary stations must be occupied by topographers for intersecting on numerous points, they should be selected with reference to this requirement. They should command an extended view, especially of points suitable for cutting in, such as hill and mountain summits, houses, churches, etc.

Wherever possible, the instrument should be accurately centered under the signal. Whenever it is necessary to set up off center the direction and distance to the signal should be carefully measured and recorded.

#### SIGNALS.

While signals should be as simple and inexpensive as possible, their form and material must depend upon the requirements and the materials at hand. In a mountainous country where the summits are treeless, simple cairns of stone 7 to 10 feet in height are employed. Where the summits are wooded it is frequently convenient to clear them, leaving a single tree to serve as a signal. In such cases it is advisable to trim the tree of branches, with the exception of a tuft at the top. Where the station is clear but with green timber easily accessible, it is advisable to make a tripod of small trees, each with a tuft at its top. In undulating and hilly country it is often necessary to erect scaffolds. These should be built of sawed lumber and framed in simple fashion. If the lines are short a pole with a flag may be set in the top. If the lines are long the tower itself may serve as a signal, in which case its upper part should be clothed in black and white cotton.

Pl. III shows a form of framed signals adapted to the treeless plains of Kansas and the rolling open hills of New England and elsewhere where observing towers are not necessary.

It is frequently necessary to raise the instrument to a considerable elevation in order to overlook surrounding obstacles. In such cases the structures for supporting the instrument should be combined with the signals, and hence they may properly be described and figured here. These observing towers should be in two parts - an interior structure, solidly built of sawed lumber if available, for the immediate support of the instrument, and a framework surrounding it and supporting a platform just below the instrument stand for the observer. The two should be separate, in order that the jarring incident to moving about on the platform may not be communicated to the instrument. Such a type of observing tower is shown in Pl. IV.

In the Sierra Nevada of California, among the sugar-pine forests, a



SIGNAL COMBINED WITH TOWER FOR OBSERVER AND INSTRUMENT.



support for the instrument is frequently obtained by sawing off the top of a high tree and setting the instrument upon the stump, 50 or 75 feet above the ground, the tree being guyed by wire cables to prevent swaying. Neighboring trees, sawed off, support the platform for the observer. Similar devices are resorted to in the forests of West Virginia, Kentucky, and Tennessee. In secondary triangulation in these regions the instrument's support in many cases is provided as above described, while the observer's platform is attached to the same tree instead of having an independent support. This is objectionable, but is often the best plan available.

In other cases it is more economical to support the instrument upon the ground, and to have openings made thru the forest upon the station hill, in the directions of the sight lines, or even to have the whole summit cleared.

#### HELIOTROPES.

It is often necessary to use more elaborate forms of signals, especially when the point observed upon is below the horizon line, so that the background, instead of being the sky, consists of forests or brown plains. In such cases resort is had to heliotropes. These are nothing more than devices for reflecting the sunlight to the observer at the instrument. The simplest form is a circular mirror with a screw hinged at the back, giving a universal motion. This is screwed into a stake or tripod over the center of the station to be observed upon, and a ray of sunlight is thrown thru a small hole in a board nailed to a stake 10 or 15 feet away and in the direction of the observer at the distant station. This form has the advantage of simplicity, as the simplest backwoodsman can manage it; and the triangulator can firmly fix all range stakes upon one visit to the station, and be sure of seeing the flash as he observes from each of the surrounding stations in turn.

Two other forms are in use—the Coast Survey type and the Steinheil. The former consists of a telescope which is provided with a screw for fastening it into any convenient support or upon the theodolite. Upon the telescope are a mirror and two rings, the axis of the rings as well as the center of support of the mirror being parallel to the line of sight of the telescope. The telescope being directed upon the observing station, the mirror is so turned as to reflect the sunlight thru the rings and necessarily to the observing station. In many cases the use of a second mirror is necessary, owing to the relative position of the two stations and the sun, and such a mirror forms a part of the outfit. This form is little used, on account of its liability to get out of adjustment. The Steinheil heliotrope is a compact little instrument which can be easily carried in a case. It consists of a small sextant mirror, the two surfaces of



which are as nearly absolutely parallel as possible. This mirror has a small hole in the center of the reflecting surface. Below this hole is a small lens in the shaft carrying the mirror, and below the lens is some white reflecting material, as plaster of Paris. The mirror is so mounted that it has four motions, two about its horizontal axis and two about its vertical axis, each of which can be separately bound or controlled by clamps or friction movements. To use the Steinheil, it is screwed into some wooden upright, as the side of a tree in such a position that the main axis carrying the lens and plaster of Paris reflector shall be parallel with the sun's rays. The observer, standing behind the mirror, receives from the rear surface of the glass a reflection of the sun, producing an imaginary sun. The mirror should not be moved until this imaginary sun, moving with it, appears to rest on the object to which it is desired to cast the flash, as the hill on which the triangulator is standing. As the surfaces of the mirror are parallel, the true reflected rays of the sun will also be cast on the object sighted to.

This instrument is in great favor, especially with the Western parties, where portability is of moment, first, because it is light and convenient to carry and use, and, second, because there are no movable parts to get out of adjustment by jarring. The latter is a serious defect in the Coast Survey instrument, since it requires frequent testing to make sure that neither of the two rings has been moved, as such movement would cause the reflections to be cast out of parallelism with the line of sight of the telescope.

The use of heliotropes presupposes the employment of men to operate them, thus increasing materially the expense of the work. Misunderstandings continually arise between the heliotropers and the observer, causing vexatious delays, and therefore their employment should be avoided whenever possible.

#### THEODOLITES FOR TRIANGULATION.

Instruments differing widely in power and degree of accuracy have been in use by the United States Geological Survey for the measurement of angles in the primary triangulation. Formerly theodolites having circles 6, 7, 8, 10, and 11 inches in diameter and reading by vernier to 10 seconds were employed, and the results were reduced and adjusted by least squares. Subsequently it appeared desirable to employ instruments of a higher class and thus obtain more accurate results, which would render unnecessary this tedious adjustment. Pursuant to this decision the use of these vernier theodolites has been largely discontinued, and theodolites having 8-inch circles, reading by micrometer microscopes, have been substituted almost universally in the primary work. (See Pl. V.)



EIGHT-INCH THEODOLITE.

—

The circle, as above stated, has a diameter of 8 inches, and is subdivided to 10 minutes. The object glass is 2 inches in diameter and its focal distance is  $16\frac{1}{2}$  inches. The telescope with the eyepiece commonly used has a power of about 30 diameters.

The circle is read by means of two microscopes placed opposite each other. Within the field of the microscope is a comb stretching over the space of 20 minutes. This comb has ten teeth, divided into two parts by a depression, each corresponding to 2 minutes. Parts of a minute down to 2 seconds are read by means of a micrometer screw moving a pair of fine threads in the field of the microscope.

#### INSTRUCTIONS FOR MEASUREMENT OF HORIZONTAL ANGLES.

The following general precautions should be observed in the measurement of all horizontal angles in the primary triangulation:

The instrument should have a stable support, which may be a stone pier, a wooden post, or a good tripod. If a portable tripod is used its legs should be set firmly in the ground.

The instrument should be protected from the direct rays of the sun by means of an umbrella or a piece of canvas like a tent fly. It should also be shielded from winds that might jar or twist either it or its support.

The foot screws of the instrument, after it is leveled for work, should be tightly clamped. Looseness of the foot screws and tripod is a common source of error, especially with small instruments.

The alidade, or the part of the instrument carrying the telescope and verniers or microscopes, should move freely on the vertical axis. Clamps should likewise move freely when loosened. Whenever either of these moves tightly the instrument needs cleaning, oiling, or adjusting.

The observer should always have a definite preliminary knowledge of the objects or signals observed. The lack of it may lead to serious error and entail cost much in excess of that involved in getting such knowledge.

Great care should be taken to insure correctness in the degrees and minutes of an observed angle. The removal of an ambiguity in them is sometimes a troublesome or expensive task.

The errors to which measured angles are subject may be divided into two classes—those dependent on the instrument used, or instrumental errors; and those arising from all other sources, which, for the sake of distinction, may be called extrainstrumental errors.

The best instruments are more or less defective, and all adjustments on which precision depends are liable to derangement; hence the general practise of arranging observations in such a manner that the errors due to instrumental defects will be eliminated in the end results.

The principal errors of this kind and the methods of avoiding their effects are enumerated below.

Measurements made with a graduated circle are subject to certain systematic errors commonly called periodic. Certain of these errors are always eliminated in the mean (or sum) of the readings of the equidistant verniers or microscopes, and both of the latter should be read with equal care in precise work. Certain other errors of this class are not eliminated in the mean of the microscope readings, and only these need consideration. Their effect on the mean of all the measures of an angle may be rendered insignificant by making the number of individual measures with the circles in each of  $n$  equidistant positions separated by an interval equal to  $\frac{360^\circ}{mn}$ , where  $m$  is the number of equidistant verniers or microscopes. Thus, if  $m = 2$ , the circle should be shifted after each measure by an amount equal to  $\frac{180^\circ}{n}$ , which, for example, is  $45^\circ$  if  $n = 4$  and  $30^\circ$  if  $n = 6$ . The degree of approximation of this elimination increases rapidly with  $n$ . The effect of errors of this class is always nil on an angle equal to the angular distance between consecutive microscopes or a multiple thereof. Other things being equal, therefore, we should expect the measures of such special angles to show less range than the measures of other angles.

Besides the instrumental errors of the periodic class, there are also accidental errors of graduation. These are in general small, however, in the best modern circles and their effect is sufficiently eliminated by shifting the circle in the manner explained in the preceding paragraph.

The effect of an error of collimation on the circle reading for any direction varies as the secant of the altitude of the object observed. The effect on an angle between two objects varies as the difference between the secants of their altitudes. This effect is eliminated either by reversing the telescope in its wyes, or by transmitting it without changing the pivots in the wyes, the same number of measures being obtained in each of the two positions of the telescope. The latter method is the better one, especially in determining azimuth, since it eliminates at the same time errors due to inequality of pivots and to inequality in height of the wyes.

The effect of the error of inclination on the circle reading for any direction varies as the tangent of the altitude of the object observed. If the inclination is small, as it may always be by proper adjustment, its effect will be negligible in most cases. But if the objects differ much in altitude, as in azimuth work, the inclination of the axis must be carefully measured with the striding level, so that the proper cor-

rection can be applied. The following formula includes the corrections to the circle reading on any object for collimation and inclination of telescope axis:

$$c \sec h + b \tan h;$$

$c$  = collimation in seconds of arc,

$b$  = inclination of axis in seconds of arc,

$h$  = altitude of object observed.

Parallax of wires occurs when they are not in the common focal plane of the eyepiece and objective. It is detected by moving the eye to and fro sidewise while looking at the wires and the image of the object observed. If the wires appear to move in the least, an adjustment is necessary. The eyepiece should always be first adjusted to give distinct vision of the cross wires. This adjustment is entirely independent of all others and requires only that enough light to illuminate the wires enter the telescope or microscope tube. This adjustment is dependent on the eye and is in general different for different persons; hence maladjustment of the eyepiece can not be corrected by moving the cross wires with reference to the objective. Having adjusted the eyepiece, the image of the object observed may be brought into the plane of the cross wires by means of the rack-and-pinion movement of the telescope. A few trials will make the parallax disappear.

When circles are read by micrometer microscopes it is customary to have them so adjusted that an even number of revolutions of the screw will carry the wires over the image of a graduation space. If the adjustment is not perfect an error of run will be introduced. This may in all cases be made small or negligible since, by means of the independent movements of the whole microscope and the objective with respect to the circle, the image may be given any required size. In making this adjustment some standard space, or space whose error is known, should be used. At least once at each station where angles are read observations should be made for run of micrometers.

Tangent and micrometer screws should move freely, but never loosely. In making a pointing with the telescope the tangent screw should always move against or push the opposing spring. Likewise, bisections with the micrometer wires should be made always by making the screw pull the micrometer frame against the opposing spring or springs.

Extraintstrumental errors may be divided into four classes—errors of observation, errors from twist of tripod or other support, errors from centering, and errors from unsteadiness of the atmosphere.

Barring blunders or mistakes, the errors of observation are in general relatively small or unimportant. With practised observers in

angular measurements, such errors are the least formidable of all the unavoidable errors, and their elimination in the end results is usually well-nigh perfect. The recognition of this fact is very important, for observers are prone to attribute unexpected discrepancies to bad observation rather than to the much more probable cause. After learning how to make good observations the observer should place the utmost confidence in them, and never yield to the temptation of changing them because they disagree with some preceding observations. Such discrepancies are in general an indication of good rather than poor work.

Stations or tripods which have been unequally heated by the sun or other source of heat usually twist more or less in azimuth. The rate of this twist is often as great as a second of arc per minute of time, and it is generally nearly uniform for intervals of ten to twenty minutes. The effect of twist is to make measured angles too great or too small according as they are observed by turning the microscopes in the direction of increasing graduation or in the opposite direction. This effect is well eliminated, in general, in the mean of two measures, one made by turning the microscopes in the direction of increasing graduation and followed immediately by turning the microscopes in the opposite direction. Such means are called combined measures or combined results, and all results used should be of this kind. As the uniformity in rate of twist can not be depended on for any considerable interval, the more rapidly the observations on an angle can be made the better will be the elimination of the twist. The observer should not wait more than two or three minutes after pointing on one signal before pointing on the next. If for any reason it should be necessary to wait longer, it will be best to make a new reading on the first signal.

The precision of centering an instrument or signal over the reference or geodetic point increases in importance inversely as the length of the triangulation lines. Thus, if it is desired to exclude errors from this source as small as a second, one must know the position of the instrument within one-third of an inch for lines a mile long or within 6 inches for lines 20 miles long. The following easily remembered relations will serve as a guide to the required precision in any case:

- 1 second is equivalent to 0.3 inch at the distance of 1 mile.
- 1 second is equivalent to 3.0 inches at the distance of 10 miles.
- 1 second is equivalent to 6.0 inches at the distance of 20 miles.
- 1 minute is equivalent to 1.5 feet at the distance of 1 mile.

The notes should always state explicitly where the instrument and signals are and give their coordinates (preferably polar coordinates) if they are not centered.

Objects seen thru the atmosphere almost always appear unsteady, and sometimes this unsteadiness is so great as to render the identity of the object doubtful. It is usually greatest during the middle of

the day, and generally subsides or ceases an hour or two before sundown. There is also frequently a short interval of quietude about sunrise, and on cloudy days many consecutive hours of steady atmosphere may occur. For the best work observations should be made **only** when the air causes small or imperceptible displacements of signals. In applying this rule, however, the observer must use his discretion. Errors of pointing increase rapidly with increase of unsteadiness, but it will frequently happen that time may be saved by counterbalancing errors from this source by making a greater number of observations. Thus, if signals are fairly steady it may be economical to make double the number of observations rather than wait for better conditions.

The best results in a triangulation are to be obtained by measuring the angles separately and independently. Thus, if the signals in sight around the horizon are in the order A, B, C, etc., the angles A to B, B to C, etc., are by this method observed separately, and whenever there is sufficient time at the observer's disposal this method should be followed.

Besides measuring single angles, it is desirable to measure independently combined angles—i. e., angles which consist of the sum of two or more single angles. Thus, supposing O to be the observing station and A, B, and C stations sighted on, the observer should measure not only the angles AOB and BOC, but the combined angle AOC. This is necessary not only because this angle may be used directly in the triangulation, but because it will be needed in forming conditions for adjusting the angles about the observing station, or the station adjustment, as it is called.

In order to secure the elimination of the errors mentioned above, the following procedure must be strictly adhered to:

Point on A and read both microscopes.

Point on B and read both microscopes.

Transit telescope and turn microscopes  $180^\circ$ .

Point on B and read both microscopes.

Point on A and read both microscopes.

Shift circle by  $\frac{180^\circ}{n}$  and proceed as before until  $n$  such sets of measures have been obtained.

Then measure the angles B to C, C to D, etc., including the angle necessary to close the horizon, in the same manner.

A form for record and computation of the results is given on page —.

When repeating instruments are used, the same procedure will be followed, except that there should be five pointings instead of one on each of A and B, the circle being read for the first pointing on A and the fifth on B, and again for the sixth pointing on B and the tenth on A.



The importance of having the measures of a set follow in quick succession must be constantly borne in mind. Under ordinarily favorable conditions an observer can make a pointing and read the microscopes once a minute, and a set of five repetitions should be made in five minutes or less.

When several stations or signals are visible and a nonrepeating instrument is used, time can be saved without material loss of precision in the angles, by observing on all the signals successively according to the following procedure, the signals being supposed in the order A, B, C, etc., as above:

- Point on A and read microscope.
- Point on B and read microscope.
- Point on C and read microscope.
- Point on A and read microscope.
- Transit telescope and turn microscopes  $180^\circ$ .
- Point on A and read microscope.
- Point on B and read microscope.
- Point on C and read microscope.
- Point on A and read microscope.

Shift circle by  $\frac{180^\circ}{n}$  and proceed as before until  $n$  such sets have been obtained.

The angles A to B, B to C, etc., read in this way may be computed as in the first method, always combining the measure A to B with the immediately succeeding measure B to A to eliminate twist. There is a theoretic objection to this process of deriving angles, founded on the fact that they are not independent, but in secondary work this objection may be ignored as of little weight.

For the 11-inch theodolite and for the 8-inch instruments made by Fauth & Co., all of which read by micrometer microscopes, four sets of measures on as many different parts of the circle will be required; and for the repeating theodolite six sets of measures will be required, all measures being made according to the procedures given above.

Under ordinary circumstances and with due care in centering, angles measured as specified above should show an average error of closure of less than 5 seconds. Under specially unfavorable conditions the number of sets of measures should be increased, care being always taken to shift the circle so as to eliminate periodic errors.

The practise of starting the measurement of an angle or series of angles with the microscopes reading  $0^\circ$  and  $180^\circ$ ,  $90^\circ$ , and  $270^\circ$ , etc., should be avoided; otherwise the errors of these particular divisions will affect many angles. In shifting the circle it is neither necessary

nor desirable to have the new positions differ from the preceding one by exactly  $\frac{180^\circ}{n}$ . A difference of half a degree either way is unimportant as respects periodic errors, and it is advantageous to have the minutes and seconds differ for the different settings.

Field notes should be clear and full. The date, place, name, and number of instrument used, and the names of observer and recorder should be recorded at the beginning of each day's work at a station. The positions of the instrument and signals observed should be defined, either by a full statement or by a reference to such, in each day's notes. The time of observations should be noted at intervals, to show that the instrument does not stand too long between pointings.

A preliminary reduction of all observations should be made in the field, in order to detect and correct gross errors. A plat of the triangulation should be made as the work progresses.

When mistakes are made in the record, the defective figures should not be erased, but simply crossed out, and an explanation furnished in the column of remarks. Great care should be taken not only to avoid "cooking" or "doctoring" notes, but to avoid suspicion thereof.

The following example of form of record is taken from the primary triangulation executed in western Kansas:

*Record of measurement of horizontal angle.*

Station: Township corner, Kansas, July 1, 1889. Fauth 8-inch theodolite No. 362, one division of micrometer head = 2 seconds.

Station.	Micr. A.			Micr. B.			Mean reading.			Angle.			Mean.
	Telescope direct.												
	Div.			Div.			°   '   "			°   '   "			
Walton.....	93	12	11.3	273	12	00.9	93	12	21.2	36	29	03.9	05.9
Newt.....	129	41	11.9	309	41	13.2	129	41	25.1				
Newt.....	129	41	15.6	309	41	12.1	129	41	27.7			08.0	
Walton.....	93	12	10.6	273	12	09.1	93	12	19.7				
	Telescope reversed.												
Walton.....	138	27	03.2	318	26	28.0	138	27	01.2				01.8
Newt.....	174	56	02.8	354	55	28.9	174	56	01.7			00.5	
Newt.....	174	56	06.2	354	55	29.5	174	56	05.7				
Walton.....	138	27	05.2	318	26	27.4	138	27	02.6			03.1	
	Telescope reversed.												
Walton.....	183	07	03.0	3	06	27.2	183	07	00.2				03.9
Newt.....	219	36	05.0	39	35	29.8	219	36	04.8			04.6	
Newt.....	219	36	08.1	39	35	29.5	219	36	07.6				
Walton.....	183	07	06.4	3	06	28.1	183	07	04.5			03.1	
	Telescope direct.												
Walton.....	228	24	28.1	48	24	22.6	228	24	50.7				04.3
Newt.....	264	53	27.4	84	53	26.1	264	53	53.5			02.8	
Newt.....	264	54	01.1	84	53	26.1	264	53	57.2				
Walton.....	228	24	29.3	48	24	22.1	228	24	51.4			05.8	
Mean of 4 combined measures $a = 36^{\circ} 29' 03''.98$													4 15''.9

$a$  Instrument over center of station.

## ORGANIZATION OF PARTIES AND PROSECUTION OF WORK.

A party for carrying on primary triangulation usually comprizes only the chief and an assistant, with the addition of a driver and a cook in case the party is living in camp. Frequently, however, it is found economical to employ a man to superintend the construction of signals. The chief of party is expected to select the stations and direct what forms of signals shall be erected, and to measure angles. In a mountainous country the selection of stations is usually a simple matter. From the summit of a mountain the chief of party may be able to select stations for considerable distances ahead and to order the erection of signals, leaving to the man employed for that purpose the erection of them. On the other hand, in a densely wooded region such as the Cumberland Plateau, where the summits have approximately the same elevation, the selection of stations is extremely difficult, requiring great ability and experience and involving an immense amount of labor. In such a region the chief of party finds it necessary to travel great distances, visit many hills, and even to climb to the summits of the highest trees, in order to select intervisible stations.

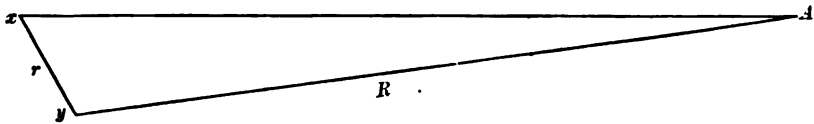
The selection of stations must be kept in advance of the reading of angles, but it is not advisable to keep it too far ahead, on account of the danger of the destruction of signals before angles have been read upon them. Therefore the chief of party finds it necessary to alternate between the two kinds of work, selecting and preparing three or four stations, then returning and measuring the angles.

When it is necessary to use heliotropes the party must be increased by one man for each such instrument employed. The proper management of such a party then calls for the exercise of much judgment on the part of the triangulator. If it is convenient for the chief of party to place each heliotrope before observing angles, and to show him where to direct his instrument, men of ordinary intelligence may be employed, and the work is one calling for time rather than for skill. Where, however, the party is moving frequently the observer and heliotropes occupying different stations nearly every day, as is possible in the dry atmosphere usually prevailing in the West, the chief of party has to arrange a schedule for each man, showing the order in which he is to occupy the stations and in what direction he is to flash from each. In this case the heliotrope must be a man having some topographic and technical skill, so that he may find his point, set up on center, and direct his flashes to the right place, besides exercising a goodly amount of judgment. A simple code of signals having been agreed upon, it becomes easy for the triangulator to let the heliotrope know that the work is completed, when he at once moves to the next designated station.

REDUCTION OF PRIMARY TRIANGULATION.

REDUCTION TO CENTER.

In case any station is occupied off center, the directions as read must first be reduced to center. In the diagram, let *x* be the point



occupied, *y* the station, *r* the distance between them, *A* the point to which the direction is laid and the angle at that point, and *R* its distance, approximately known. Then, from the relations between the sides and the angles of the triangle,

$R : r :: \sin x : \sin A,$   
 $\sin A = \frac{r \sin x}{R},$  and  $A$  (in seconds) =  $\frac{r \sin x}{R \sin 1''}$   
correction in seconds of arc.

The following example taken from the triangulation in Kansas will illustrate the form of effecting this reduction. The references are to the diagram on page 59:

Reduction to center of station at Walton  $\Delta$ , Kansas.

[See explanation: Appendix No. 9, page 167, U. S. Coast and Geodetic Survey report for 1882.]

Distance, inst. to center = 0'.48 log = 9.6812  
log feet to meters = 0.5160  
Distance, inst. to center log meters = 9.1652 = log *r*.

Direction.	x to n 7°.	x to o 73°.	x to p 105°.	x to q 185°.	x to r 273°.	x to s 306°.
Log sin angle.....	9.0859	9.9806	9.9843	8.9403	9.9994	9.9080
Colog distance.....	5.9321	5.9182	6.4228	6.2434	6.0079	6.2514
Log <i>r</i> .....	9.1652	9.1652	9.1652	9.1652	9.1652	9.1652
Colog sin 1".....	5.3144	5.3144	5.3144	5.3144	5.3144	5.3144
Correction to direction.....	9.4976 0".31	0.3784 2".39	0.8873 7".71	9.6633 0".46	0.4869 3".06	0.6390 4".36

Correction to angle *a* = *n* to *o* - 0.31 + 2.39 = + 2.08  
*b* = *o* to *p* - 2.39 + 7.71 = + 5.32  
*g* = *n* to *p* - 0.31 + 7.71 = + 7.40  
*c* = *p* to *q* - 7.71 - 0.46 = - 8.17  
*d* = *q* to *r* + 0.46 - 3.06 = - 2.60  
*e* = *r* to *s* + 3.06 - 4.36 = - 1.30  
*h* = *q* to *s* + 0.46 - 4.36 = - 3.90  
*f* = *s* to *n* + 4.36 + 0.31 = + 4.67

## SPHERICAL EXCESS.

The angles are measured on a spherical surface, and the sum of the three measured angles of each triangle should equal  $180^\circ$  plus the spherical excess. The latter, however, needs to be computed and subtracted from the sum of the angles only for the purpose of testing the accuracy of closure of the triangle, as in the reduction the angles are treated as plane angles. When the area of the triangle is large the spherical excess in seconds ( $E$ ) should be computed by the equation,

$$E = \frac{S}{r^2 \sin 1''},$$

where  $S$  is the area of the triangle in square miles, and  $r$  the radius of curvature of the earth in miles. When the triangle (being within the United States) has an area less than 500 square miles,  $r$  may be assumed as constant, and the spherical excess may be obtained by dividing the area in square miles by 75.5.

## STATION ADJUSTMENT.

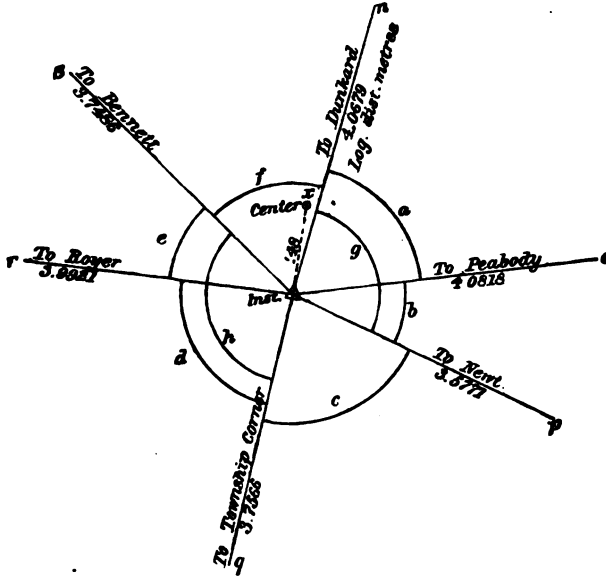
The next step is the adjustment of the angles about the observing station, or the station adjustment, as it is called. Referring to the diagram, which represents the angles read at Walton station, in Kansas, it is seen that eight angles were measured, as follows:

*Record of angles measured at Walton, Kansas.*

	Obs. angle.			Station adjust- ment.	Correc- tion to center.	Angles locally adjusted and reduced to center.		
	o	'	"			o	'	"
<i>a</i> Dunkard—Peabody.....	65	45	28.37	+ .51	+2.08	65	45	30.96
<i>b</i> Peabody—Newt.....	31	47	58.50	+ .52	+5.32	31	48	04.34
Sum.....	97	33	26.87			97	33	35.30
<i>g</i> Dunkard—Newt (meas.).....	97	33	28.39	- .49	+7.40	97	33	35.30
Difference.....	-1.52					00.00		
<i>d</i> Township corner—Royer.....	87	44	57.41	- .56	-2.60	87	44	54.25
<i>e</i> Royer—Bennett.....	34	00	03.35	- .56	-1.30	34	00	01.49
Sum.....	121	44	60.76			121	44	55.74
<i>h</i> Township corner—Bennett.....	121	44	59.05	+ .59	-3.90	121	44	55.74
Difference.....	+1.71					00.00		
<i>f</i> Bennett—Dunkard.....	61	09	26.17	+ .02	+4.67	61	09	30.86
<i>g</i> Dunkard—Newt.....	97	33	28.39	- .49	+7.40	97	33	35.30
<i>c</i> Newt—Township corner.....	79	32	06.25	+ .02	-8.17	79	31	58.10
<i>h</i> Township corner—Bennett.....	121	44	59.05	+ .59	-3.90	121	44	55.74
Sum.....	359	59	59.86			360	00	00.00
	-0.14					00.00		

Of these  $a + b$  should =  $g$ ,  $d + e$  should =  $h$ , and  $g + c + h + f$  should  $360^\circ$ . Thus are formed in this case three conditions affecting eight unknown quantities. The method by which are found the correc-

tions which fulfil these conditions is that known as the method of least squares, for a full discussion of which, showing its application to triangulation, see "The Adjustment of Observations," by T. W. Wright and J. F. Hayford, pp. 180-260, (New York, D. Van Nostrand, 1906). It is unnecessary to explain the theory of this method, but it is desirable to show how it is applied in the class of cases under consideration,



which can best be done by tracing a case thru. There are here three equations of conditions, as follows:

- (1)  $a + b - g - 1''.52 = 0;$
- (2)  $d + e - h + 1''.71 = 0;$
- (3)  $f + g + c + h - 0''.14 = 0;$

in which the letters represent, not angles, as in the diagram, but unknown corrections to the angles. The coefficient of each of these corrections is unity. Arrange them in tabular form, the letters at the top referring to the equations, thus forming what is called a table of correlates. Now multiply each coefficient by itself and every other in the same horizontal line and sum them. Three normal equations result as follows:

	<i>w</i>	<i>y</i>	<i>z</i>	
<i>a</i>	1			1
<i>b</i>	1			1
<i>c</i>			1	
<i>d</i>		1		
<i>e</i>		1		
<i>f</i>			1	
<i>g</i>	-1		1	
<i>h</i>		-1	1	

1	+3.00 <i>w</i>	-1.00 <i>z</i>	-1''.52 = 0
2		+3.00 <i>y</i>	-1.00 <i>z</i> + 1''.71 = 0
3	-1.00 <i>w</i>	-1.00 <i>y</i>	+4.00 <i>z</i> - 0''.14 = 0

These three equations involving three unknown quantities are then solved by elimination, with results as follows:

$$w = +.515.$$

$$y = -.562.$$

$$z = +.023.$$

These values can now be substituted in the table of correlates, columns 1, 2, 3; the algebraic sum of lines  $a, b, c, d$ , etc., giving corrections to the angles  $a, b, c, d$ , etc.

	1	2	3	Corrections to angles.
$a$	+.515			+.515
$b$	+.515			+.515
$c$			+.023	+.023
$d$		-.562		-.562
$e$		-.562		-.562
$f$			+.023	+.023
$g$	-.515		+.023	+.023
$h$		+.562	+.023	+.492
				+.585

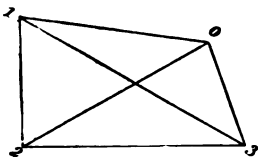
FIGURE ADJUSTMENT.

The measurement of the angles having been executed by instruments and methods much better than the needs of the map require, it is not ordinarily necessary to make any figure adjustment further than an equal distribution of the error of each triangle among the three angles. Still, as the necessity for a more elaborate adjustment may arise, a description of the method of applying the least squares to adjustment of geometric figures in triangulation is here given, with a simple example of its application.

Each geometric figure in a system of triangulation is composed of a number of triangles. The measured angles of each triangle should equal  $180^\circ$  plus the spherical excess. Each triangle, therefore, furnishes an equation of condition, which is known as an angle equation. The number of angle equations in any figure is equal to the number of closed triangles into which it can be resolved. But since certain of these are a consequence of the others, the number of angle conditions which it is desirable to introduce is less than the number of triangles.

The number of angle equations in any figure is equal to the number of closed lines in the figure plus one, minus the number of stations. Thus, in a closed quadrilateral, the number of angle equations is  $6 + 1 - 4 = 3$ .

There is another class of conditions, known as side equations, which can be best explained by reference to a figure. In the diagram, suppose the figure 0, 1, 2, 3 to represent the projection of a pyramid, of which 1, 2, 3 is the base and 0 the apex. A geometric condition of such figure is that the sums of the logarithmic sines of the angles about the base, taken in one direction, must equal the similar sums taken in the other direction—i. e., the product of the sines must be equal. In



the present case,  $\log \sin 0, 1, 2 + \log \sin 0, 2, 3 + \log \sin 1, 3, 0$  should equal  $\log \sin 1, 2, 0 + \log \sin 2, 3, 0 + \log \sin 0, 1, 3$ .

The number of side equations which can be formed in any figure is equal to the number of lines in the figure, plus 3, minus twice the number of stations in it, or  $l + 3 - 2n$ . In a quadrilateral,  $6 + 3 - 8 = 1$ .

The numerical term in each angle equation is the difference between the sum of the observed angles on the one hand and  $180^\circ$  plus the spherical excess on the other. This is positive when the sum of the observed angles is the greater and vice versa. The coefficients of the unknown corrections are in each case unity, unless weights are assigned.

The numerical term in each side equation is the difference between the sums of the logarithmic sines, taken in the two directions. The coefficients of the unknown corrections are the differences for one second in the logarithmic sines of the angles.

The method of making up and solving these equations and applying the corrections to the angles can best be shown by means of an example. That here given is the simplest case involving both angle and side equations, namely, the case of a quadrilateral. The method of forming correlatives and normal equations, and their solution, is similar to that employed in station adjustment, and therefore the details are omitted.

In the equations of conditions and correlatives the angles are designated by directions to which the corrections are finally applied. Thus the angle of  $3, 0, 2$  is designated  $-3/0 + 2/0$ , the sign  $-$  being given to the left-hand and the sign  $+$  to the right-hand direction.

## EXAMPLE OF FIGURE ADJUSTMENT BY LEAST SQUARES.

		Observed angles.		
		°	'	"
(a) ...	3-0-1	120	39	14.781
	0-1-3	21	26	17.806
	1-3-0	37	54	37.180
		180	00	09.767
	Spherical excess	= - 0.148		
		Closure error		
		+ 9.619		
(b) ...	0-1-2	81	52	51.222
	1-2-0	62	22	38.500
	2-0-1	35	44	45.861
		180	00	15.583
		- 0.189		
		Closure error		
		+ 15.304		
(c) ...	1-2-3	91	28	38.000
	2-3-1	28	95	10.360
	3-1-2	60	26	33.416
		180	00	21.776
		- 0.234		
		Closure error		
		+ 21.542		
(c) ...	2-3-0	65	59	47.540
	3-0-2	84	54	28.920
	0-2-3	29	05	59.500
		180	00	15.960
		- 0.193		
		Closure error		
		+ 15.767		



*Side equation.*

[Taking 0 as the pole.]

	Angle.	Log sines of spherical angle	Tabular difference for 1"	Corrections to log sines.	Corrected log sines of spherical angles.	Spherical excess.	Log sines of plane angles.
(d)	0.1.2.....	9.9956249.7	+3.0	- 25.0	9.9956224.7	-.063	9.9956224
	0.2.3.....	9.6869340.0	37.9	-127.9	9.6869212.1	-.065	9.6869210
	1.3.0.....	9.7884705.9	27.0	- 1.2	9.7884704.7	-.050	9.7884703
	Sum.....=	29.4710295.6			29.4710141.5		29.4710137
	1.2.0.....	9.9474437.5	- 11.0	- 59.4	9.9474378.1	-.063	9.9474378
	2.3.0.....	9.9607184.9	9.4	- 77.7	9.9607107.2	-.064	9.9607107
	0.1.3.....	9.5628859.2	53.7	-203.0	9.5628656.2	-.049	9.5628653
	Sum.....=	29.4710481.6			29.4710141.5		29.4710137
	From above...	29.4710295.6					
	Difference...	00.0000186.0			000.0		0000.

$$(d) \dots\dots\dots 0 = +186.0 - 3.0 \left(\frac{1}{4}\right) + 03.0 \left(\frac{1}{7}\right) - 37.9 \left(\frac{2}{3}\right) + 37.9 \left(\frac{4}{4}\right) - 27.0 \left(\frac{4}{4}\right) + 27.0 \left(\frac{3}{3}\right) \\ - [-11.0 \left(\frac{4}{4}\right) + 11.0 \left(\frac{4}{5}\right) - 9.4 \left(\frac{4}{5}\right) + 9.4 \left(\frac{3}{5}\right) - 53.7 \left(\frac{7}{7}\right) + 53.7 \left(\frac{7}{7}\right)].$$

*Equations of condition.*

$$(a) \dots\dots\dots 0 = + 9''.619 - \frac{2}{3} + \frac{1}{5} - \frac{2}{3} + \frac{1}{5} - \frac{2}{3} + \frac{1}{5} \\ (b) \dots\dots\dots 0 = +15.394 - \frac{1}{3} + \frac{1}{5} - \frac{1}{3} + \frac{1}{5} - \frac{1}{3} + \frac{1}{5} \\ (c) \dots\dots\dots 0 = +15.767 - \frac{1}{3} + \frac{1}{5} - \frac{1}{3} + \frac{1}{5} - \frac{1}{3} + \frac{1}{5}$$

Collecting terms in (d) and dividing thru by 100 so as to avoid dealing with large numbers.

$$(d) \dots\dots\dots 0 = +1.86 + .507 \left(\frac{1}{4}\right) + .030 \left(\frac{1}{7}\right) - .489 \left(\frac{2}{3}\right) + .379 \left(\frac{4}{4}\right) - .270 \left(\frac{4}{4}\right) \\ + .176 \left(\frac{4}{5}\right) + .110 \left(\frac{4}{5}\right) + .094 \left(\frac{4}{5}\right) - .537 \left(\frac{7}{7}\right).$$

*Table of correlatives.*

Direction.	a.	b.	c.	d.
0-1	-1	-1	.....	+.507
0-2	.....	+1	-1	-.489
0-3	+1	.....	+1	+.176
1-0	+1	+1	.....	.....
1-2	.....	-1	.....	+.110
1-3	-1	.....	.....	-.270
2-0	.....	-1	+1	.....
2-1	.....	+1	.....	+.030
2-3	.....	.....	-1	+.094
3-0	-1	.....	-1	.....
3-1	+1	.....	.....	-.537
3-2	.....	.....	+1	+.379

Forming the normal equations in the usual manner, we have:

	"				
(a).....	0 = + 9.619	+6.000	+2.000	+2.000	-0.598
(b).....	0 = +15.394	+2.000	+6.000	-2.000	-1.076
(c).....	0 = +15.767	+2.000	-2.000	+6.000	+0.950
(d).....	0 = - 1.860	-0.598	-1.076	+0.950	+1.054

Solving, we find the following values:

$$\begin{aligned} a &= + 1.900 \\ b &= - 4.386 \\ c &= - 5.208 \\ d &= + 3.059 \end{aligned}$$

Substituting the values of  $a$ ,  $b$ ,  $c$ ,  $d$ , in the table of correlatives.

Direction.	a.	b.	c.	d.	Correction to each direction.
?	-1.900	+4.386		+1.551	+4.037
?		-4.386	+5.208	-1.496	-0.674
?	+1.900		-5.208	+0.538	-2.770
?	+1.900	-4.386			-2.486
?		+4.386		+0.336	+4.722
?	-1.900			-0.826	-2.726
?		+4.386	-5.208		-0.822
?		-4.386		+0.092	-4.294
?			+5.208	+0.288	+5.496
?	-1.900		+5.208		+3.308
?	+1.900			-1.643	+0.257
?			-5.208	+1.159	-4.049

	Observed angles.	Corrections.	Corrected spherical angles.	Sph. excess.	Plane angles.
	° ' "	" "	° ' "	"	° ' "
3.0.1	120 39 14.781	-3.308-2.486	120 39 08.986	-.049	120 39 08.94
0.1.3	21 26 17.806	-4.037+0.257	21 26 14.026	-.049	21 26 13.98
1.3.0	37 54 37.180	+2.726-2.770	37 54 37.136	-.050	37 54 37.08
			180 00 00.148	-.148	180 00 00.00
0.1.2	81 52 51.222	-4.037-4.294	81 52 42.891	-.063	81 52 42.83
1.2.0	62 22 38.500	-4.722-0.674	62 22 33.104	-.063	62 22 33.04
2.0.1	35 44 45.861	+0.822-2.486	35 44 44.194	-.063	35 44 44.13
			180 00 00.189	-.189	180 00 00.00
1.2.3	91 28 38.000	-4.722-4.049	91 28 29.229	-.078	91 28 29.15
2.3.1	28 05 10.360	-5.496-2.726	28 05 02.138	-.078	28 05 02.06
3.1.2	60 26 33.416	-0.257-4.294	60 26 28.865	-.078	60 26 28.79
			180 00 00.232	-.234	180 00 00.00
2.3.0	65 59 47.540	-5.496-2.770	65 59 39.274	-.064	65 59 39.21
3.0.2	84 54 28.920	-3.308-0.822	84 54 24.794	-.064	84 54 24.73
0.2.3	29 05 59.500	+0.674-4.049	29 05 56.125	-.065	29 05 56.06
			181 00 00.193	-.193	180 00 00.00

## COMPUTATION OF DISTANCES.

In each triangle, starting with the base line, there is known at least one side and the three angles. The remaining sides are computed by the well-known proportion of sides to sines of opposite angles, or, express mathematically,  $a = \frac{b \sin A}{\sin B}$ . In this computation distances should be used in meters, and seven-place logarithms should be employed.

The following is an example of the correction of the angles and the computation of the sides of triangles, taken from the work in Kansas:

*Correction of angles and computation of sides of triangles.*

Station.	Angles locally adjusted and reduced to center.	$\frac{1}{3}$ error.	Plane angles.	Log sines.
	<i>D</i> <i>S</i> <i>H</i>	<i>H</i>	<i>D</i> <i>S</i> <i>H</i>	
Township corner.....	36 29 04.0	+ .5	36 29 04.5	0.2257704
Newt.....	63 58 56.2	+ .6	63 58 56.8	9.9533952
Walton.....	79 31 58.1	+ .6	79 31 58.7	9.9927124
	179 59 58.3			
	Error = -1.7			

Log dist. Newt-Walton.....	3.5771611
Log sin Newt.....	9.9533952
a. c. log sin Township corner.....	0.2257704
Log dist. Township corner-Walton.....	3.7563267
Log dist. Newt-Walton.....	3.5771611
Log sin Walton.....	9.9927124
a. c. log sin Township corner.....	0.2257704
Log dist. Township corner-Newt.....	3.7956439

## COMPUTATION OF GEODETIC COORDINATES.

The next step is the computation of the latitude and longitude of the stations and the azimuth or direction of the lines connecting them. Initially, the latitude and longitude of some point are determined by astronomic observations, and this point is connected with the triangulation. The azimuth, or angle with a south line, of a line connecting this point with some station in the triangulation is also determined by astronomic observations. These, with the observed angles and the computed distances between the stations, form the data from which the latitudes and longitudes of the stations and the azimuths of the lines connecting them are computed. The difference in latitude between two adjoining stations is obtained from the following equation, based upon the Clarke spheroid:

$$-dL = K \cos a' B + K^2 \sin^2 a' C + (\delta L)^2 D - hK^2 \sin^2 a' E,$$

in which

$dL$  is the difference in latitude;

$K$ , the distance between the stations in meters;

$a'$ , the fore azimuth of the line connecting them, measured around clockwise from the south thru the west;

$h$ , the first term;

$\delta L$ , the approximate difference in latitude, being the sum of the first two terms;

$B$ ,  $C$ ,  $D$ , and  $E$ , constants derived from the dimensions and figure of the earth (these are given for various latitudes in tables in Appendix 9 of United States Coast and Geodetic Survey Report for 1894).

The difference in longitude is obtained by means of the formula,

$$dM = \frac{K \sin a' A'}{\cos L'},$$

in which

$dM$  is the difference in longitude;

$L'$ , the newly determined latitude;

$A'$ , a constant, from tables; and the others as above.

The azimuths at the ends of a line differ from each other because of the convergence of the meridians. That first determined is known as the fore azimuth, the other as the back azimuth. All azimuths are measured from the south point around clockwise.

The back azimuth is computed from the formula,

$$-da = dM \frac{\sin \frac{(L + L')}{2}}{\cos \frac{1}{2} dL},$$

in which

$M$  is the longitude of the first station;

$L$ , the latitude, and

$L'$ , the latitude of the second station.

The constants used are those of the Clarke spheroid of 1866.

These formulas are derived and explained in Appendix No. 9, Report United States Coast and Geodetic Survey for 1894.

The following are examples of the use of the formulas, taken from the triangulation in New Mexico:

*Examples of computation of geodetic coordinates.*

(1)	Azimuth $a$ :	Nell—Chusca.	° ' "		
	Spherical angle:		159	29	08.728
			120	54	13.980
			<hr/>		
	Azimuth $a'$ :	Nell—Zuni.	38	34	54.748
	$\delta a + 180^\circ$		179	50	02.124
			<hr/>		
	Azimuth ( $a$ ):	Zuni—Nell.	218	24	56.872

## GEODETIC COORDINATES.

LATITUDE.				LONGITUDE.			
	°	'	"		°	'	"
L	35	25	13.443	Nell.	M	108	37 24.925
d L	-17	47.546		Geo. Pos. No. 5.	d M	+	17 15.360
L'	35	07	25.927	Zuni.	M'	108	54 40.285
				Geo. Pos. No. 6.			
Computation for latitude:				Computation for longitude:			
log K	4.6236305			log K	4.6236305		
" B	8.5111933			" sin a'	9.7949286		
" cos a'	9.8930500			" A'	8.5092394		
				" sec L'	0.0872944		
log (I)	3.0278738			Corr. for diff. arc & sine	-15		
log K²	9.24726			log (V)	3.0150914		
" C	1.25606			d M	10357.360		
" sin² a'	9.58986						
log (II)	0.09408			Computation of azimuth:			
log D	2.3679			log (V)	3.015091		
" [I+II]²	6.0568			" sin $\left(\frac{L+L'}{2}\right)$	9.761522		
log (III)	8.4247			" sec $\left(\frac{d L}{2}\right)$	0.000001		
log E	6.0124			log (VI)	2.776614		
" K² sin² a'	8.8371			da	- 597".876		
" (I)	3.0279				- 9' 57".876		
log (IV)	7.8774			Azimuth check.			
"							
(I)	1066.286+						
(II)	1.242+						
(III)	.026+	[I+II]	1067.528				
(IV)	.008-	log "	3.0283792	Check:			
-dL	1067.546+	" [I+II]²	6.0567584	Spher. angle at			

Computation of Azimuth  $a$ , in Book —, page —.  
 Spherical angle and distance =  $K$ , in Book —, page —, Triangle No. —.  
 — Station; Computed by —.

(2)

		°	'	"
Azimuth $a$ :	Chusca—Nell.	339	21	40.150
Spherical angle:		25	11	38.601
Azimuth $a'$ :	Chusca—Zuni.	4	33	18.751
$d a + 180^\circ$		179	57	25.650
Azimuth ( $a$ ):	Zuni—Chusca.	184	30	44.401

## GEODETIC COORDINATES.

LATITUDE.			LONGITUDE.		
	°	' "		°	' "
L:	35	53 06.746	Chusca.	M:	108 50 14.518
d L	-	45 40.818	Geo. Pos. No. 4.	dM	+ 4 25.768
L'	35	07 25.928	Zuni.	M'	108 54 40.286
			Geo. Pos. No. 6.		
Computation for latitude:			Computation for longitude:		
log K	4.	9280539	log K	4.	9280539
" B	8.	5111594	" sin a'	8.	8999280
" cos a'	9.	9986260	" A'	8.	5092394
			" sec L'	0.	0872944
			Corr. for diff. arc & sine	-	129
log (I)	3.	4378393	log (V)	2.	4245028
log K²	9.	85610	dM	+265''.	768
" C	1.	26435			
" sin² a'	7.	79982	Computation of azimuth:		
log (II)	8.	92027	log (V)	2.	424503
log D	2.	3703	" sin $\left(\frac{L+L'}{2}\right)$	9.	764002
" [I+II]²	6.	8757	" sec $\left(\frac{d L}{2}\right)$	0.	000009
log (III)	9.	2460			
log E	6.	0214	log (VI)	2.	188514
" K² sin² a'	7.	6559	d a	-	154''.
" (I)	3.	4378		-2'	34''.
log (IV)	7.	1151			
(I)	2740.	560+	Azimuth check:		
(II)		.083+		°	' "
(III)	.176+			218	24 56.872
(IV)	.001-			184	30 44.401
-d L	+2740.	818			
			log [I+II]	2740.	643
			log [I+II]²	3.	4378525
			" [I+II]²	6.	875705
			Check:	33	54 12.471
			Spher. angle		
			at Zuni	33	54 12.469

Computation of Azimuth a, in Book 67, page 4.

Spherical angle and distance=K, in Book 64, page 12, Triangle No. 3.

Station: Computed by H. M. W.

When the lines are not more than 20 miles in length the equation for latitude may be simplified without appreciable error by dropping the last two terms.

## TRAVERSE LINES FOR PRIMARY CONTROL.

In a level country, especially if covered with forests, it is very expensive to carry on triangulation, and in some cases practically impossible to do so. Under such circumstances traverse lines afford the only means of obtaining adequate control for maps.

A traverse line consists of a series of direction and distance measurements. Each course, as the direction and the accompanying distance are called, depends upon the one immediately preceding it, and a continuous chain is thus formed. Traverse lines are largely used in topographic work proper for making minor locations. The primary traverse differs from these only in the fact that it is much more elaborately executed.

The initial point of a primary traverse must be located either by triangulation or by astronomic determinations. The end of the line should, if possible, be a point similarly well located. The line should, if practicable, follow a railroad, in order to obtain the easiest possible grades, and thus avoid errors incident to slope.

The instruments used should be a 20- or 30-inch transit, a 300-foot steel tape, a spring balance, two flag poles, a thermometer, and a watch.

The party should consist of one chief to act as transitman, one recorder, two tapemen, and a flagman. At each station the transitman should proceed as follows:

The telescope should be set on the rear flag, both verniers read, the telescope transited, pointed on the front flag, and both verniers read. The circle should be shifted and the same angle remeasured with the telescope reversed.

Along a railroad the operation of measuring should be conducted as follows:

The front tapeman should attach to the front end of the tape a 20-pound tension with the spring balance. He should make a check mark on the rail or drive a tack or nail in the tie. The distance which he records should be checked by the transitman and by at least one other member of the party. The rails should be counted as a check upon the distance.

Along highways or across open country the tape should be kept level. The plumb bob should be used on steep slopes to bring the tape vertically over the point. Tape lengths should be marked on the measuring board with the marking needle. Where slopes are too steep for leveling with the 300-foot tape a shorter tape should be used. The temperature of the tape should be taken every hour during progress of the work.

Observations for azimuth should be made at the close of each day's work, if possible, and azimuth stations should be not more than 10 miles apart except on long tangents.

Where the line traversed is very crooked the instrument should be fitted for observation with a solar attachment, and this should be used at least twice each day, weather permitting, in addition to the observations on Polaris.

Careful notes should be taken, with a description of the starting point of the line, at the beginning and ending of each day's work. The location of each railroad station should be noted, also each mile-post, switch, wagon road, stream, land or county line cross, and connection made with corners of the public-land surveys. Permanent marks should be placed at each end of the line and also at prominent junction joints, from which other primary-control lines may be started.

Lines of traverse exceeding 100 miles in length should be reduced by computation. The distances should be corrected for error of tape and for temperature and slope, and should be reduced to sea

level, in the same manner as described in treating of the reduction of base lines, in case these corrections are of sufficient amount to affect the length appreciably upon the map.

The courses should be corrected for convergence of meridians. Then, commencing at the initial point, the latitude and departure of each station, one from another, should be computed in feet. The sum of the latitudes converted into seconds of latitude gives the difference in latitude, and the sum of the departures converted into seconds of longitude gives the difference in longitude.

Short lines of traverse may be platted with minute-reading protractors, but in this platting the utmost care should be exercised.

#### PRECISE AND PRIMARY ELEVATIONS.

For the control of elevations level lines of three orders of accuracy are employed:

(1) Precise levels, which are run from the seacoast or from previously established precise benches to distant fields of work. These are of the highest order of accuracy obtainable.

(2) Primary levels, which are run across quadrangles or around their boundaries for the immediate purpose of furnishing primary points for heights. Under the law at least two primary bench marks should be established in each land-office township, and an equal number in regions where no land surveys have been made.

(3) Flying levels, which are run over traverse lines for the purpose of locating contours.

The instruments used in precise and primary levels are in all respects similar to those employed by the Coast and Geodetic Survey, which are figured and described in the United States Coast and Geodetic Survey Reports for 1899 and 1903.

In connection with these descriptions are general instructions for the use of these instruments in running precise lines, which differ only in minor details from the instructions followed by the United States Geological Survey.

In regions where secondary triangulation is practicable heights may be measured with the plane table directly from datum points established by the primary leveling and be carried thruout the work by means of this instrument.

When the control of the map is effected by means of primary traversing, such traverse should be accompanied by a level line.



## CHAPTER V.

### SECONDARY TRIANGULATION AND TRAVERSE AND STADIA MEASUREMENTS.

#### SECONDARY TRIANGULATION.

The work of making secondary locations by intersection is done mainly by plane table. The use of the theodolite for this purpose is restricted to those cases where but little of this kind of location can be effected, and where, therefore, it seems scarcely worth while to prepare plane-table sheets.

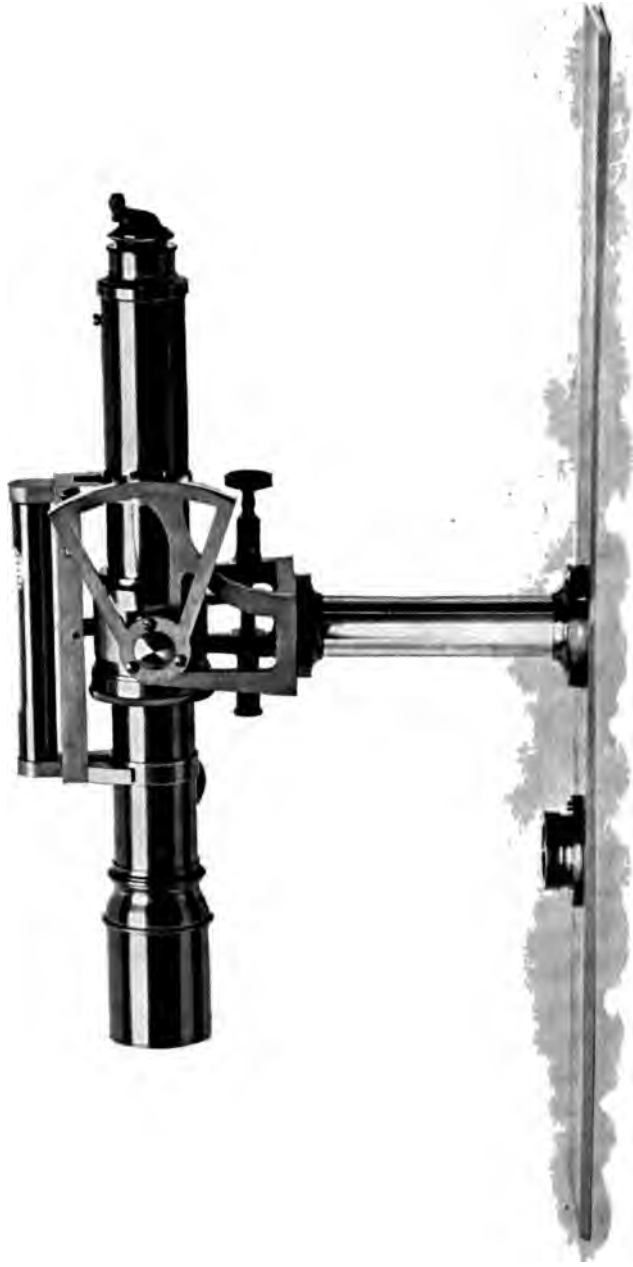
By means of the primary triangulation, three or four points are usually located upon each plane-table sheet. Within this primary triangulation, and depending upon it, are then located a large number of points, either by intersection, by traverse, or by both methods, forming a geometric framework upon which the sketching of the map depends.

Location by intersection should be carried as far as practicable—that is, all points capable of being located in this manner should be so placed, in order to afford the most ample control possible for the traverse lines, by which the intervening areas are to be filled in, it being understood that location by intersection is more accurate and more rapid than location by traverse, and consequently in every way more economical.

#### THE PLANE TABLE.

Much misapprehension exists regarding the character and application of the plane table. This apparently arises from the fact that it is little known. It is applicable to all kinds of country, to all methods of work, and to all scales. It is a simple, direct, and economical instrument, rendering possible the making of a map directly from the country as copy, and doing away with elaborate notes, sketches, photographs, etc., which not only are more expensive, but produce inferior results. (See Pl. VII.)

The plane table consists of a board upon which is fastened a sheet of drawing paper. This board is mounted upon a tripod, which, in the more elaborate forms of the instrument, possesses great stiffness and stability. It should be capable of being leveled, of being turned in azimuth, and of being clamped in any position. On the paper is produced directly in miniature a representation of the country.



TELESCOPIC ALIDADE.



When set up at different places within the area in process of being mapped, any edge of the board must always extend in the same direction—that is, a certain edge of the board must always be set at the same angle with the north and south line. This is called orienting the board.

Directions are not read off in degrees and minutes, but platted directly on the paper. The instrument used for this purpose is known as the alidade, and consists of a ruler with a beveled edge, to which are attached for rough work two raised sights, and for the higher class of work a telescope, whose axis is fixed parallel to the edge of the ruler. This telescope carries also a delicate level and a vertical arc for the measurement of angles in the vertical plane, from which relative heights are obtained. The method of using this instrument is extremely simple in principle and not difficult in practise.

(Pl. VI.)

The making of locations from intersections obtained by means of the plane table requires that the instrument have the utmost stability consistent with lightness and portability. It demands an alidade equipped with a telescope of considerable power and good definition. In short, it is necessary that the plane table be in every respect of the best modern type in order that the highest degree of accuracy may be attained. Various forms of plane-table movement have been in use, including the heavy and cumbersome but stable movement employed by the United States Coast and Geodetic Survey, and the light but unstable movement used by the same organization in its less important work. At present a table is in general use which was invented by Mr. W. D. Johnson, of the United States Geological Survey, which combines, in a remarkable degree, the elements of stability, lightness, and facility of operation. (See Pl. VII and fig. 2.) The movement is essentially an adaptation of the ball-and-socket principle, so made as to furnish the largest practicable amount of bearing surface. It consists of two cups, one set inside the other, the inner surface of one and the outer surface of the other being ground so as to fit exactly. The inner cup is in two parts, or rather consists of two rings, one outside the other, one controlling the movement in level and the other that in azimuth. From each of these rings a screw projects beneath the movement, and upon each of these screws is a nut by which it is clamped. There is no tangent screw for either the leveling or the azimuth motion, as none is required. The movement is sustained by a light hard-wood tripod with split legs. The boards used generally accommodate a full-sized atlas sheet, but they necessarily differ in size, owing to the different scales of field work adopted. The largest board used for this movement holds an atlas sheet on a scale of 1:45000, and is 24 by 36 inches in size.

The kind of paper for plane-table sheets is of great importance, especially in intersection work, as paper which expands and contracts differently in different directions under varying conditions of moisture will easily produce errors of magnitude in the work. It matters little if the paper contracts and expands, provided it does so uniformly in all directions, but all paper is made with more or less fiber, and accordingly expands and contracts more in one direction than in another. To counteract this, two thicknesses of paper are used—

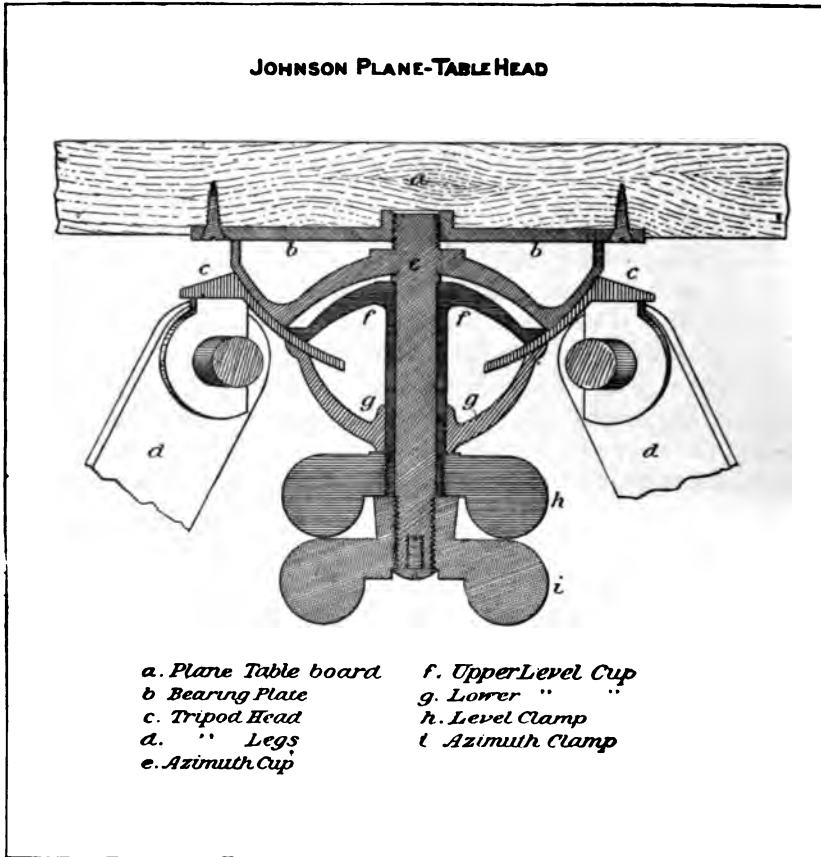
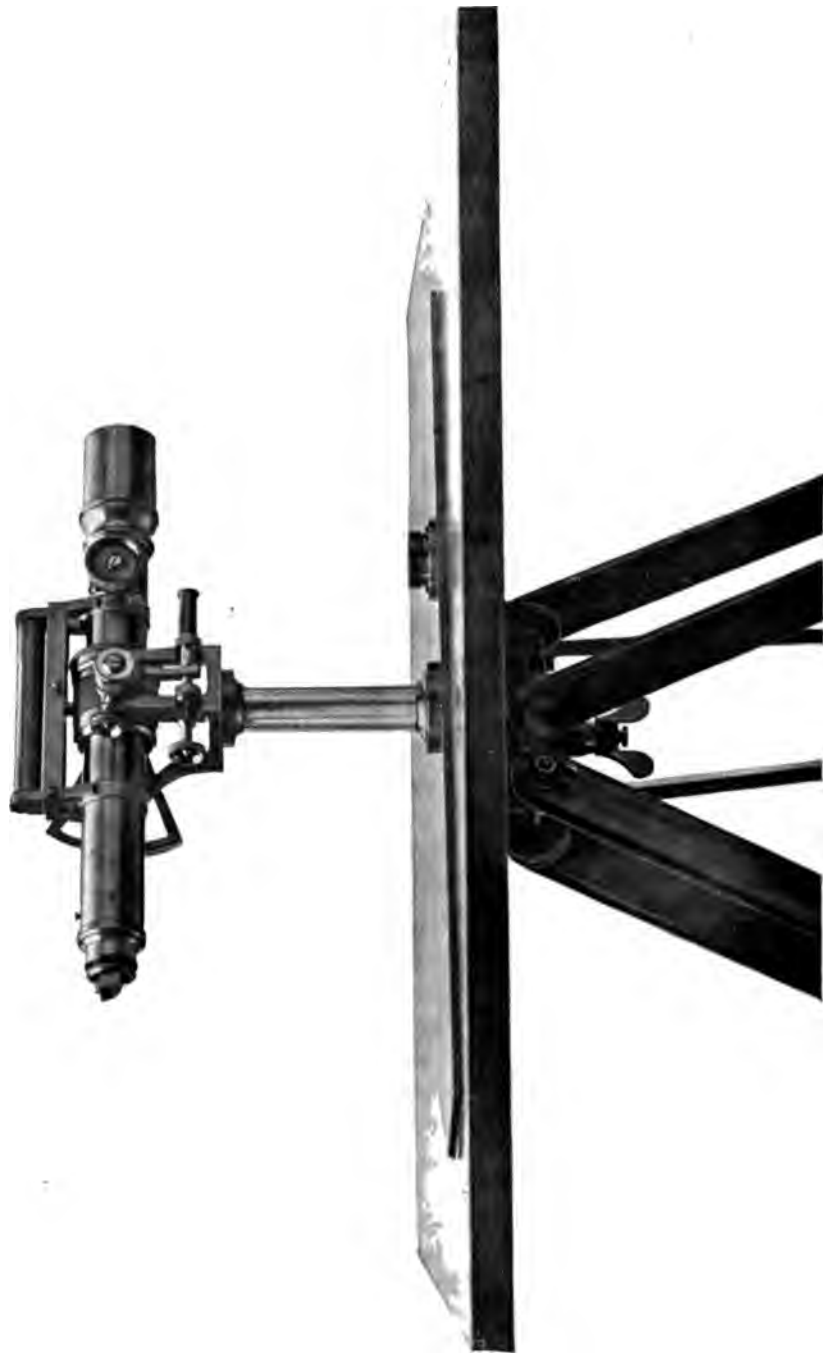


FIG. 2. Johnson plane-table movement (section).

preferably that known as Paragon paper—mounted with the grain of one sheet at right angles to that of the other, and with cloth between the layers. In sheets so prepared it has been found that there is practically no distortion, even under the most severe tests.

The board is generally made of seasoned white pine, from one-half to five-eighths of an inch thick, with cleats across the ends, fastened in such way as to allow the body of the board to contract and expand freely, thereby preventing warping. Into the corners of this board



JOHNSON PLANE TABLE AND TELESCOPIC ALIDADE.



and on the edges at points halfway between the corners are set female screws for holding the paper to the board. At corresponding points in the plane-table sheet are punched holes half an inch in diameter, thru which pass screws with broad heads fitting into the female screws in the board. The holes in the paper, being larger than the screws, allow the paper to expand or contract freely when the screws are loose; when tightened, the broad heads of the screws bind the paper firmly in place.

## THE ALIDADE.

The alidade used with the plane table (see Pls. VI, VII) consists of a ruler of brass or steel 18 inches to 2 feet in length, sometimes graduated upon a chamfered edge to suit the scale of work, and carrying upon a column a telescope having a focal distance of 12 to 15 inches and a power of about 15 diameters. It has a vertical arc of 60 degrees, reading by vernier to single minutes, and a delicate level upon the telescope, the latter turning in a sleeve for the adjustment of vertical collimation. The arc is graduated in one direction only, reading from 0 to 60 degrees, and the reading is approximately 30 degrees when the telescope is horizontal. This is to obviate any possible error in the sign of the correction for level.

Upon the plane-table sheet is constructed a projection on the scale of the field work, and upon that are platted such of the primary points as fall on the sheet, each plane-table sheet being made to correspond to an atlas sheet. These primary points are first occupied by the plane tabler.

The instrument is set over one of these stations, leveled, and clamped in level. The ruler edge of the alidade is then laid upon the line connecting this station with a neighboring one on the sheet, and the table turned in azimuth until the other station is upon the vertical wire in the telescope. The instrument is then oriented and, after clamping in azimuth, is ready for work. Keeping the ruler upon the occupied station on the sheet, the telescope is then turned upon other objects which it is desirable to locate, and lines are drawn in turn toward them. The instrument is then taken up and moved to a second station, where it is again set up, leveled, and oriented, as before. A sight is then taken, and a line drawn in the direction of each point sighted from the first station. The intersection of each pair of sight lines is the true position of the corresponding point on the map. In this way station after station is occupied by the plane table, and numerous points are located by intersection. If possible, each point thus located should be intersected from at least three stations, in order to verify its location.

Any point located on the map may be used afterwards as a station. For the location of points which have not been fixt many different



methods have been proposed, all of which are graphic solutions of the three-point problem. Probably the simplest and easiest of application is the following, which requires that three located points be visible, and that their distances be approximately known:

Set up, level, and clamp the instrument in level and azimuth, after orienting it as nearly as possible. Set the alidade successively on the three points and draw lines from their platted positions. Then the desired location will be found at a distance from each of these lines, measured at right angles to it, proportional to the distance on the ground between the point occupied and the known point. Thus the point of intersection of these lines at right angles to the lines of sight is the desired position. Were the distances from the known points to the occupied point accurately known, its position on the paper would be obtained at the first trial. Practically, however, two or three approximations are commonly found necessary.

Another method often used is as follows:

Fasten upon the plane-table board, which necessarily has not yet been oriented, a piece of tracing linen. Assume a point on this linen to represent the station, take sights upon and draw lines to all located points within the range of vision, and then, loosening the linen from the board, move it about over the map until these sight lines fall upon the proper points on the map. Then prick thru the position of the station from the linen to the map underneath. This location should then be tested by sighting from the point thus found to the various objects, to see if the sight lines fall upon the points as marked on the map. This method has the advantage of permitting the use of all located points that may be in sight, but it is not regarded as so accurate as the method first described.

In case one line of sight upon the required station has been obtained, that sight line may be utilized in making the location as follows, by resection: Having leveled the table, place the alidade upon this sight line already drawn, with the telescope pointing toward the object from which the sight was taken. Then turn the table in azimuth until the telescope falls upon this point, and clamp it. The table is now oriented, but the position of the present station is unknown further than that it is on this line. Then select some station whose direction makes a wide angle with this line, and move the alidade until the cross wire falls upon this selected station, while the ruler at the same time is upon the representation of the station on the map. The ruler will then cross the sight line at the point desired. By way of check, repeat the process with another station or located point. For this purpose a point in a suitable direction is valuable in proportion to its proximity.

Using the instrument as described above, the topographer locates all possible points. Then visiting in turn such of them as he finds

necessary, perhaps a dozen or twenty, he locates by intersection points all over the sheet in as great number and as well distributed as possible, and with special reference to the needs of the traverse men, who will come after him and whose work will be located by means of his determinations. All this work must be done with the utmost nicety and precision. The setting of the alidade upon the station must bisect the needle hole by which it is marked and the lines of direction must be drawn with a sharp-pointed pencil.

The necessity for precision will be recognized when it is understood that any error introduced in the early part of the plane-table triangulation will be not only perpetuated but increased many times over as the work progresses, and as soon as an error becomes appreciable it produces difficulties and uncertainties in making locations, which may lead to embarrassing delays and ultimately require that all the work be repeated.

#### MEASUREMENT OF ALTITUDES.

While making horizontal locations of points with the plane table, their heights also, relative to that of the point occupied, must be measured. This is done by means of the vertical arc of the alidade and the level on the telescope. Pointing upon the object whose relative height is to be measured, the telescope must first be brought to a horizontal position and the index error read. The telescope is then raised or deprest to the point and the reading obtained. This reading of the index error must be done for each point, as the table can not be leveled with sufficient accuracy and can not be expected to maintain its level so as to dispense with it. Knowing the horizontal distance to the point and the angle of elevation and depression, the difference in height is obtained by the solution of a right-angled triangle, thus:

$$h = d \tan a,$$

$h$  being the difference in height,  $d$  the distance, and  $a$  the angle of elevation or depression. This distance is then to be corrected for curvature of the earth and for refraction by the atmosphere. The correction for the former is obtained with sufficient accuracy by the following empirical rule: The curvature in feet equals two-thirds the square of the distance in miles. It is always positive in sign, whatever may be the sign of the difference in height.

Refraction is an uncertain and variable quantity. It is usually greatest at morning and night and least at midday. It is greater the nearer the line of sight is to the ground. Often in desert regions it is excessive. It is usually assumed as one-seventh the curvature and is negative.

Tables for the solution of vertical-angle work are used. These give differences in height for all angles and distances which should be employed, with corrections for curvature and refraction.

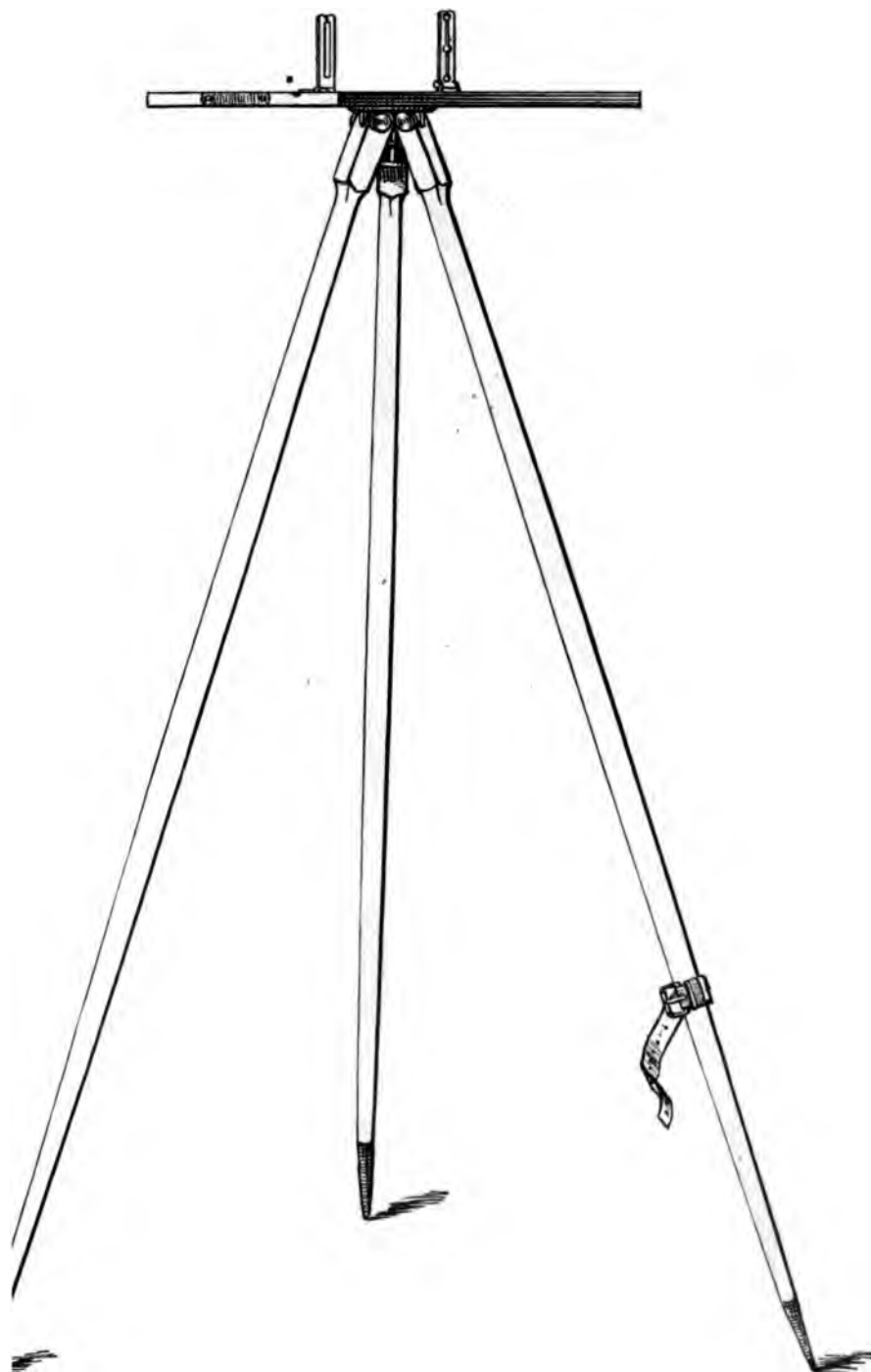
Differences of height should not be measured at greater distances than 10 miles if it can be avoided. An error of 1' in the measurement of the angle is at this distance about 15 feet, while the uncertainty of refraction in such a length of line is necessarily great.

#### TRAVERSE WORK.

As stated under the head of traverses for primary control (p. 67), a traverse line consists of a series of direction and distance measurements depending upon one another. These lines should be connected wherever possible with triangulation points in order to check up accumulated errors. If it were practicable or economical to carry on all the work of location by intersection, this would be the most accurate and on most accounts the best way to effect it, but it is only in limited localities, such as high-mountain regions where bold topographic forms predominate and where there is little or no culture, that the method of intersection is practicable for locating all necessary points. It is probable that in nine-tenths of the area of the United States it will be found necessary to locate the details of topography, culture, and drainage by means of traverse lines. In different parts of the country the relative extent to which the two methods can be applied depends upon various circumstances, principally the amount of relief of the surface and the prevalence of forests. Thus on the Atlantic plain, which is densely covered with forest and is practically level, it is necessary to use the traverse method exclusively, including even the primary control. Passing from this as an extreme case, thru rolling and hilly country, to the high, sharp mountains of the West, the triangulation method becomes more and more applicable, while the traverse method finally becomes used but little except in the details of roads and other cultural features.

For executing traverse work various instruments have been in use for measuring directions and distances. For direction there have been used theodolites of various forms and prismatic compasses, and for distances the stadia and the wheel.

All secondary traverse work should be done with plane tables upon which the directions and distances are platted directly. The Johnson plane table, with telescopic alidade, may be used for this work. When so used, a compass should be set on the edge of the board or of the alidade, for orienting, as orientation by backsighting is not sufficiently accurate.



TRAVERSE PLANE TABLE AND RULER ALIDADE.

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## TRAVERSE PLANE TABLE.

Another plane table convenient for this purpose is of the simplest possible form, consisting of a board about 15 inches square, into one edge of which is set a narrow box containing a compass needle 3 inches in length. (See Pl. VIII.) This table is supported by a tripod of light construction, without leveling apparatus, the leveling of the instrument being effected with sufficient accuracy by the tripod legs. A single screw fastens the board to the tripod head, and the adjustment in azimuth is made by simply turning the board with the hand. It is held in place by friction. The table is adjusted in azimuth, or oriented, by means of the compass needle; that is, it is turned until the needle rests opposite the zero marks in the compass box, and is thus always made approximately parallel to itself, provided the magnetic declination remains constant.

The alidade consists of a brass ruler, 6 to 12 inches long, with folding sights. The edge of the ruler is graduated to facilitate platting of distances. Ordinary drawing paper backed with cloth is used for plane-table sheets, and is attached to the board by thumb tacks.

Distances may be measured by stadia, by tape, or by counting the revolutions of a carriage wheel. When traversing is done along roads, as is commonly the case, the distance is frequently measured by counting the revolutions of a wheel, usually one of the front wheels of a buggy or buckboard. For counting the revolutions, various automatic devices have been in use.

A traverseman is generally assigned a tract of country within which he is instructed to run traverses of all public roads and of such private roads as appear to be necessary in order to control the entire tract. If practicable, he is furnished with the positions of the points located within his tract, properly platted on his plane-table sheet, or, if these can not be furnished, with such descriptions of them as are necessary to enable him to recognize them and close his lines upon them or connect with them by triangulation. He is furnished with a horse and buggy or buckboard, traverse plane table, and aneroid. He has no rodman, but is expected to sight natural objects. Setting up his instrument at his initial station, he levels it roughly by means of the tripod legs, orients it by turning the table until the compass needle is on the zero marks in the compass box, and then, marking a point on the paper to represent his initial station and placing his alidade upon it, he points it to an object selected as his second station and draws a line in that direction. Driving along the road, he passes the point sighted at, noting the distance to it by a count of the revolutions of the wheel, and the height as recorded by the aneroid, and passes on, selecting some point from which he can see the point

sighted at. There he stops, sets up his table as before, orients it, and sights upon the same signal which he sighted from his initial station. He plots the distance to the signal along the sight line from his initial station; then from the location of the signal as thus established he plots his second station by the distance measurement and the reverse of the observed direction. In this way the work progresses, a hundred stations or more being occupied in the course of the day. In this work one should aim to make as few stations and to take as long sights as possible consistent with accuracy. Bends of the road between stations can be sketched with all needful accuracy.

During the progress of the work all points off the line which are capable of being located by intersection must be located by sights taken from stations, and special care must be taken to connect them with the points located by the secondary triangulation, in order to afford as many checks as possible to the accuracy of the traverse line.

Traverse lines should close with but trifling error—1 per cent being as great an error as should be permitted—and all errors of closure should be shown. No line should be arbitrarily closed on the traverse sheet.

The traverseman should sketch or locate all country houses, and should note all road intersections and all railroad crossings, specifying by simple conventions whether over, under, or grade crossing. He should similarly describe all stream crossings, distinguishing fords, ferries, and bridges.

#### MEASUREMENTS OF ALTITUDES IN CONNECTION WITH TRAVERSE WORK.

##### VERTICAL ANGLES.

Height measurements in connection with traverse lines are effected in one of two ways—either by vertical angles with the telescopic alidade or by the use of the aneroid.

In regions where little or no secondary triangulation can be done, it becomes necessary to accompany certain of the traverse lines by profiles determined by vertical angles. Such profiles should be surveyed at intervals of 4 or 5 miles where the contour interval is 20 feet, and at intervals of 8 or 10 miles where it is 50 feet.

The plan of the traverse is run precisely as above sketched, except that often a rodman is employed. In running the profile, which is done coincidently with the plan, the points sighted for elevation may be the same as those used for the plan. If a rodman is employed, the target on the rod should be set at the height of the instrument, to simplify record and computation.

It must not be understood, however, that it is at all necessary that the survey of the profile should establish the height of all the points located by the traverse. The profile should give the eleva-

tions of all valleys and summits and of all road crossings. The line should be carried forward and these points measured by as few and as long lines of sight as possible. Often the roof of a house will furnish a datum point for use for a mile or two. Indeed, in an open, settled country the line can frequently be carried forward continuously by using housetops as targets.

The reduction of the profile must keep pace with the field work, so that on arriving at a check point the amount of the error may be shown at once. If this is not more than one-fourth or one-fifth of the contour interval, it is not considered as of material account. If, however, it reaches half a contour interval, the work should be examined, and if the error be not discovered the line should be resurveyed.

The heights as determined should be written in ink on the plane-table sheet in their proper places.

## THE ANEROID.

In some traverse work heights are measured with aneroids. The aneroid consists of a vacuum box of thin corrugated metal, which is compressed by an increase and expanded by a decrease in the pressure



FIG. 3.—Aneroid.



FIG. 4.—Works of the aneroid.

of the atmosphere. A train of mechanism magnifies this trifling movement enormously and moves an index upon a graduated dial. This dial is graduated to feet of elevation and also to inches of barometric pressure. (See figs. 3 and 4.) Several sizes of aneroids are made; that having a diameter of  $2\frac{1}{2}$  inches is on the whole found the most satisfactory.



Owing mainly to its extreme delicacy the aneroid is a very uncertain instrument. It should be used differentially only, and for small differences in height and small intervals of time. Its indications should be checked by reference to known elevations whenever opportunity is afforded during the day, and at the beginning and end of each day's work.

On commencing work the movable scale on the aneroid should be set at the known height of the starting point and a note made of its reading on the inch scale. Elevations should then be read directly from the scale of feet. The heights of all points along the line of traverse that will be required in making the contour sketch should be read and written upon the traverse. Every depression and elevation, road crossing, etc., should thus be measured. There is, however, no necessity for reading the aneroid at every station in the traverse; that would merely encumber the work with a mass of useless data.

Upon reaching a check point, comparison should be made with the indications of the aneroid. If the difference is considerable—more than a contour interval—the error should be distributed backward along the line in proportion to distance. If it is small, it may be neglected.

In all this work notebooks are not required except as a convenient form of carrying paper on which to make the trifling computations required. The plane-table sheets comprize all the records necessary. The work, as it progresses, criticizes itself by its closures in position and elevation, and wherever necessary is revised immediately.

#### ORGANIZATION OF PARTIES AND DISTRIBUTION OF WORK.

Secondary triangulation, traversing, measuring of heights, and sketching are commonly carried on by one party, consisting of the chief, who directs all operations and does most of the sketching; an assistant who carries on the secondary triangulation, selected as possessing special fitness for that work; and one, two, or more assistants who are engaged in traversing, the number depending upon the rapidity with which the country can be sketched relative to the rate at which the traversing progresses. If possible, the different items of work of such a party should follow one another in a certain order. The secondary triangulation should be done first, in order that the traversemen may be furnished with positions and heights for locating and checking their traverse lines. The traversing should follow, in order that all the control may be furnished to the chief of party for his use in sketching. This order requires that the members of the party be scattered over a considerable area, and if they are living in camp it necessitates their remaining away from it a considerable part of the time, or else that a large amount of traveling be done in order

to reach camp at night. Where they are not living in camp, the most economical disposition is to scatter them at various places within their fields of work. In any case constant communication must be had between the chief of party and his assistants, in order that they may work in accord.

#### STADIA MEASUREMENTS.

Under certain circumstances it is found advisable to use the stadia method for measuring distances in place of the wheel. This is the case where lines are to be run without reference to roads, and consequently where the wheel can not be employed with advantage. It has been used, too, in southern Louisiana, where peculiar methods of work imposed by the nature of the topography have made its employment economical. Any instrument carrying a telescope may be used for the stadia or telemeter method of measuring distances. To the reticule of the telescope are added two or more fixed horizontal wires placed at certain distances apart. A rod or board subdivided to suit the interval between the wires and painted in glaring colors forms part of the outfit. When this rod is set up at a distance from the telescope, that distance is ascertained from the number of subdivisions of the rod which are included between the wires of the telescope, the value of each division of the rod being known. All alidades are equipped with stadia wires. These wires are three in number, the intervals between them being equal. The rods are 14 feet in length and hinged so as to close to 7 feet. The intervals on the rods are 1 foot each. For convenience, the wires in the telescope should be so spaced that when the rod is at a distance of 100 feet the space between the two extreme wires will subtend 1 foot on the rod. At a distance of 1,400 feet, therefore, this space will subtend the entire length of the rod, while at a distance of 2,800 feet two adjacent wires in the telescope will subtend the entire length of the rod. Distances less than 100 feet are estimated by means of the fractional part of a foot on the rod which is included between the wires. The distances are read off on the rod by the surveyor at the instrument.

In measuring distance on slopes correction must be made to reduce the inclination measured to horizontal distance. Where the slope is slight, it is not regarded as necessary to make this reduction, especially where there are frequent points for checking and correcting the line.

The rod may be used also for measurement of the profile of the traverse line. For this purpose a point should be marked upon it at the same height as the telescope of the instrument and vertical angles taken to this point.

The foregoing matter applies most directly to maps on scales ranging from 1 to 8 miles to an inch and covering large areas of country. It applies also, tho less fully and directly, to maps on larger scales and those made for specific purposes. Among the latter class may be mentioned maps of mining districts, of watersheds, for drainage purposes or water supply, of railroad and canal lines, of parks, town sites, etc. In making such maps, whose scales are expressed in hundreds of feet to an inch instead of miles to an inch, we deal with inches instead of feet in both horizontal and vertical measurements, and consequently greater accuracy must be obtained as expressed in units of the ground surveyed, but in units of the map the standard of accuracy is unchanged. An error of one-fiftieth of an inch on the map is as large as should be permitted. The enlarged scale and reduced area under survey permits the use of certain instruments which are not economical for smaller scales, and also permits more extended use of certain others. The use of the stadia instrument in connection with the plane table here becomes economical, and it should be used extensively. After executing a primary triangulation the work should be relegated to the plane table, with which such secondary triangulation may be effected as seems expedient. Then the stadia comes into play, and, using one, two, or more rodmen, as may be economical, the topographer will locate points by direction and distance measurements from his station, reaching out perhaps one-fourth of a mile in all directions and measuring heights by vertical angles on the rod at the same time that he locates the horizontal positions of the points. In this way he will locate points which control the contour or points on the contours directly, as seems expedient. If the country is nearly level, it may be best to do the latter, but if there is much relief he will find it more expedient to locate only controlling points and to sketch the contours with reference to them.

From one station he will pass to another, and so cover the country mainly by stadia locations.

This is by far the most accurate, thoro, and rapid method of making detailed maps of small areas.

In making a location map of a railroad or a canal line, the method is necessarily varied to some extent. The line itself is measured with accuracy with a tape, and should be platted on the plane table as measured, and will thus serve as a continuous base line. The topography of either side should be taken by stadia, two rodmen being employed, one on either side of the line.

## CHAPTER VI.

### SKETCHING, OFFICE WORK, AND PROJECTIONS.

#### SKETCHING.

Since sketching is by far the most important part of the work of map making, it should be done by the most competent man for this work in the party—as a rule, by its chief. Besides the fact that he is presumably the best sketcher in the party, there is another reason for requiring that he should execute the sketching—he is held responsible for the work, not only for the quality of the sketching but also for the accuracy and sufficiency of the control, and in the sketching of the map he has the best possible opportunity for examining into the condition of the control and of remedying any weaknesses.

Upon completing the secondary triangulation, the traverse work, and the measurement of heights—in short, the control—within an area, which may be large or small according to convenience, but preferably should comprize a quarter sheet, he should cause all this control to be assembled on one sheet. The traverse lines with all points located from them should be adjusted to the secondary locations, and all measurements of height should be platted upon this skeleton, thus presenting in complete form all the control within the area. With this sheet upon a sketching board the chief of party should go over the ground, sketching the drainage, culture, and forms of relief, and generalizing the features to suit the scale of his map. The relief should be sketched in actual continuous contours directly from the country as copy, so that upon leaving the sketching stations the only work remaining to complete the map will be inking and lettering. In heavy country, however, where the contours follow one another closely, it will often be sufficient to put in at the stations only a part of the contours—every fifth one, for instance—in order to economize time in the field. Stations for sketching may be selected with the utmost freedom. An exact location is unnecessary. Any point on or off the road which affords an outlook will serve. As a rule, frequent stations should be made, and one should not attempt to sketch at great distance unless the conditions are favorable, as in a country of large, bold features. It may be necessary to travel over all the roads that have been traversed and to climb many hills in order to sketch the entire area satisfactorily. On the other hand, in a different region the entire area

may be sketched by a limited amount of travel or from a few elevated points. In a low country of small features much travel will be required, as these details must be sketched from near points. In a bold country of high relief, which may be sketched entirely from a few points, care must be exercised in the selection of sketching stations. From a great altitude the lower details will be dwarfed and will measurably disappear, while from low points the relations, forms, and masses of the greater elevations can not be properly seen. In such a country stations at different elevations must be selected in order to see all parts of the country to the best advantage. The extreme summits will prove of little service as sketching stations.

Sketching is artistic work. The power of seeing topographic forms in their proper shapes and proportions and of faithfully transferring these impressions to paper is one of the most difficult acquirements to obtain. The difficulty is increased by the necessity of expressing form by means of continuous contour lines at fixed intervals. This work involves knowledge of the elements of structural geology and good judgment in applying them.

Whatever its scale may be, every map is a representation reduced from nature, and consequently there is more or less generalization. It is therefore impossible to make any map an accurate, faithful picture of the country it represents. Moreover, the smaller the scale the higher must be the degree of generalization, and the further must the map necessarily depart from the original.

Now, it is in this matter of generalization that the judgment of the topographer is most severely tested. He must be able to take a broad as well as a detailed view of the country; he must understand the meaning of its broad features, and then must be able to interpret details in the light of those features. Only such a man is competent to make just generalizations and to decide what details should be omitted and what preserved, and, where details are omitted, what to put in their places in order to bring out the dominant features.

It is not possible to define the degree of detail which maps should represent. The limit commonly given—that imposed by the scale of the map—is not always the best. In representing country which has little plan or system, such as moraines or sand dunes, it is well to work in as much detail as the scale will bear; but where the country shows system in its structure, to which the minor detail is subordinate, the omission of some of this detail will give greater prominence to the larger features. The amount of detail thus omitted must necessarily be left to the judgment of the topographer, but no more should be omitted than is necessary to give full expression to the general features of the country.

### SCALE OF FIELD WORK.

The scale on which the field work is executed is generally larger than that on which the maps are to be published, because the drawing in the field is necessarily coarser than is possible in the office. The field scale is commonly greater than the publication scale; that is, plane-table sheets which are to be published on the scale of 1:62500 are made in the field on the scale of 1:48000 and those to be published on the scale of 1:125000 are made in the field on the scale of 1:96000.

### OFFICE WORK.

The office work of the topographers consists in the reduction and transfer of the work from plane-table sheets to the original maps. The reduction is usually effected by photography, this method having been found the most accurate and economical. It is often the practise simply to complete the plane-table sheet by inking and lettering it, making it serve as the original sheet. On many accounts this practise is advisable.

The original sheets are to serve as the original record of the work and as manuscript for publication. To answer these purposes they are made complete in all respects. Every original sheet should contain within itself all matter which is to be engraved or placed on record.

The method by which the map is to be published should have much influence upon the preparation of the original map. In case it is to be photolithographed or photo-engraved, it must be drawn by a skilled draftsman on a scale much larger than that of publication. If it is to be engraved, however, it is unnecessary to waste the time of a skilled draftsman upon it, as the engraver requires merely clearness and legibility.

### PROJECTIONS.

The projection almost universally used is the polyconic.

The construction of a projection for a limited area is a simple matter, but requires care and accuracy and the use of the best drafting instruments.

Tables for this projection are published by the United States Coast and Geodetic Survey, which are applicable to all scales of map, the measurements given being the absolute lengths on the earth's surface, which must be reduced to the scale of the map. Tables are published in Bulletin No. 234 of the United States Geological Survey, which are directly applicable to certain scales without reduction.

First draw a line down the middle of the sheet. Lay off on this line the length of the several projection spaces in latitude by taking from the projection table for the scale the length of, say, 5 minutes of latitude

and lay it off repeatedly, thus establishing the points of intersection of parallels at each 5 minutes with the middle meridian. Thru these points draw lines across the sheet at right angles to the middle meridian, using beam compasses for this purpose. Lay off on these lines the dm's corresponding to the latitude for  $2^{\circ} 30'$  and  $7^{\circ} 30'$  from the middle meridian on each side, and at these points erect short perpendiculars. On these lay off the dp's corresponding to the dm's, and thru the points thus obtained draw and ink the projection lines.

For other areas the process is similar, but when a large area, such as that of the United States, is to be projected the mechanical difficulties greatly increase.

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[Bulletin No. 307.]

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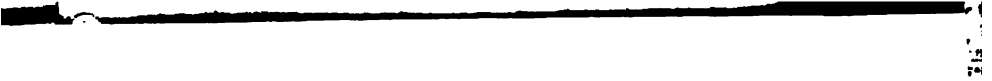
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